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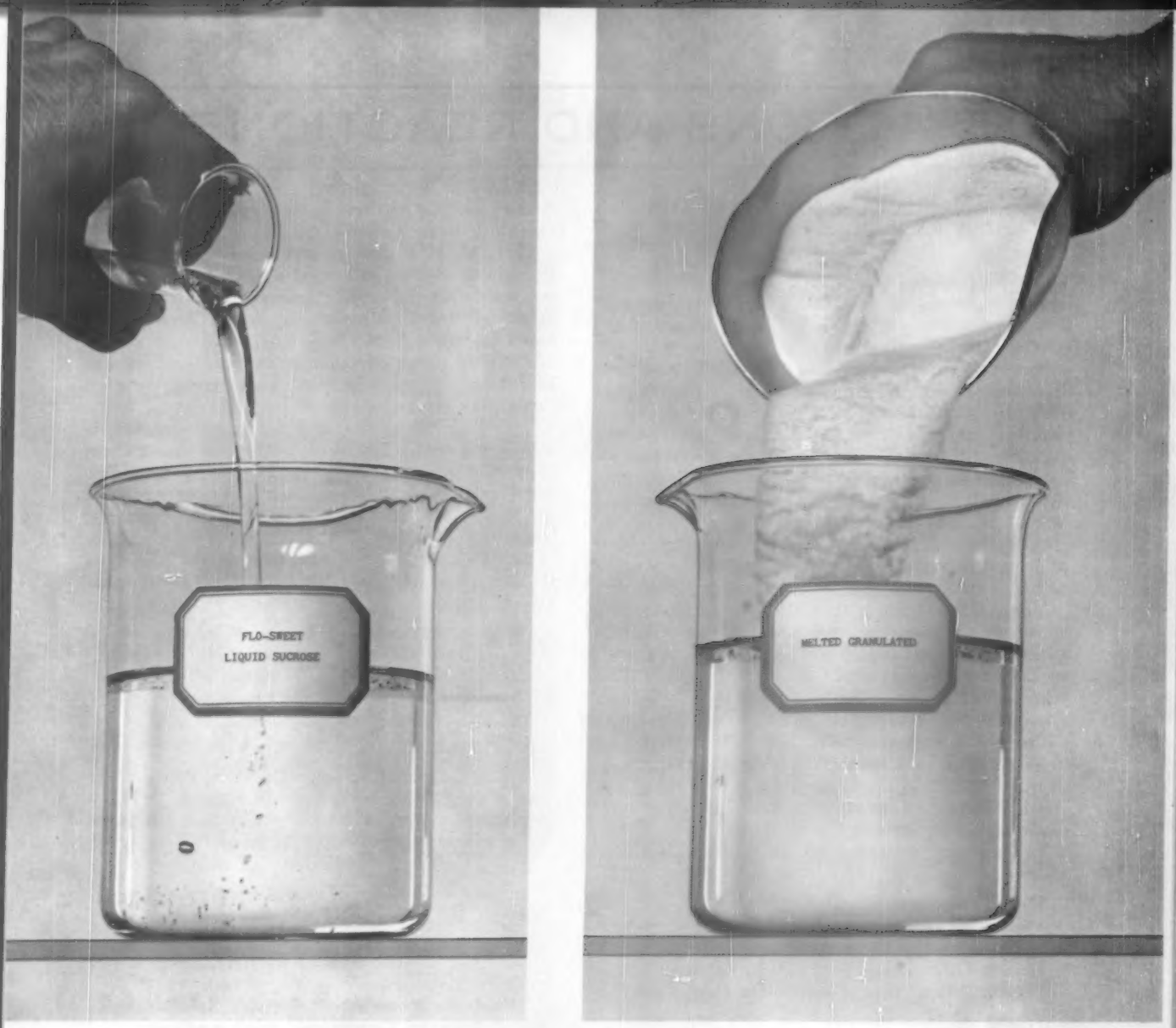

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IFT ACTIONS AND REACTIONS...

FOOD PROTECTION COMMITTEE AND INDUSTRY LIAISON PANEL REPORT FOR 1956

In 1950, the Food Protection Committee was organized as a committee under the Food and Nutrition Board of the National Research Council to act as an advisory and consulting group to both governmental agencies and private industries. The Food Protection Committee is a completely independent group of scientists comprising pharmacologists, toxicologists, physiologists, food technologists, and experts from related disciplines. It is solely responsible for its decisions and conclusions but is given financial support by industrial concerns, independent laboratories, and consultants, each of which appoints a representative to the Liaison Panel. The Panel also includes non-voting members who are representatives of trade associations, governmental agencies, and scientific and technical societies.

Many of the activities of the Food Protection Committee are the result of requests from industry transmitted through the Industry Committee which acts as an Executive Committee of the Liaison Panel. Once a year, a general meeting has been held in Washington, attended by the entire Panel, the Food Protection Committee and its subcommittees. The 1956 meeting was in Washington on December 13 under the joint chairmanship of Dr. William J. Darby, Chairman of the Food Protection Committee, and Dr. F. N. Peters, Chairman of the Industry Committee of 1956.

Reports were presented by the following subcommittees of the FPC:

Food Technology—Dr. D. B. Hand
Pesticides—Dr. G. C. Decker
Toxicology—Dr. J. M. Coon
Dietary Carcinogenesis—Dr. P. R. Cannon
"Practical Equivalent of Zero"—Dr. H. E. Carter

During the year, the FPC published or made ready for publication a number of reports, including:

"The Use of Chemical Additives in Food Processing"
"Grouping of Raw Agricultural Commodities for Residue Comparisons and the Simplification of Applications for Tolerances"
"The Relation of Surface Activity to the Safety of Surfactants in Foods"
"Safe Use of Pesticides in Food Production"

A statement dealing with chelating agents is in preparation. Study of chemicals in packaging materials has proceeded and it is expected that a report will be forthcoming in 1957. The committee has also considered the subjects of antibiotics and has decided that there is no need for special activity in this direction.

In addition, the Committee has just issued for publication a statement entitled, "What We Know About Possible Relationships Between Cancer and Food Additives." The Executive Secretary, Dr. Paul E. Johnson, published an article in the September issue of *Cereal Science Today* entitled "Chemical Additives in Foods." A similar article appeared in the December issue of the *Journal of Home Economics.*

The Ad Hoc Subcommittee on "The Practical Equivalent of Zero" presented a brief report. It was concluded that there could be no general level established, but that the "toxicological trivial" levels of different materials should be set by some mathematical relationship to the minimum deleterious level. It was believed not necessary and undesirable to use the word "zero" in the term.

The Chairman of the 1957 Industry Committee is Dr. C. J. Krister, E. I. du Pont de Nemours & Company, Wilmington, Delaware. Information regarding membership in the Liaison Panel can be obtained from Dr. Krister, Dr. Paul E. Johnson, Executive Secretary, Food Protection Committee, National Research Council, 2101 Constitution Avenue, Washington 25, D. C., or from the other members of the 1957 Industry Committee:

Dr. L. B. Parsons, Lever Brothers Company
Dr. F. D. Smith, Monsanto Chemical Company
Dr. E. J. Matson, Abbott Laboratories
Dr. J. J. Thompson, Kellogg Company
Dr. R. J. Sumner, Anheuser-Busch, Inc.
Dr. F. N. Peters, The Quaker Oats Company

Acknowledgment is given to Dr. F. N. Peters, Chairman of the 1956 Industry Committee and to Dr. O. R. Alexander, American Can Company, for their assistance in the preparation of this report.

L. E. Clifcorn
IFT Liaison Representative to
Food Protection Committee
Food and Nutrition Board
National Research Council

What We Know About Possible Relationships Between Cancer and Food Additives

Statement of the Food Protection Committee Food and Nutrition Board

In the development of our knowledge of nutrition, primary emphasis has been given to deficiencies that may occur in our diets and to ways of safeguarding against them. Along with the application of such knowledge a notable reduction in dietary deficiency diseases has occurred. With decreasing need in the United States for emphasis in this direction, more effort has been devoted to the investigation of positive factors in foodstuffs that might be detrimental to health.

A group of conditions broadly termed "degenerative diseases" has assumed major importance as causes of illness and death in recent decades. The causes of these conditions are under intensive investigation, and especial emphasis is being placed in the role of environmental factors.

Casual relationships between environmental factors and human disease have long interested scientists.

(Continued on page 8)

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(Continued from page 2)

Indeed, knowledge of such relationships underlies most advances in preventive medicine. It is not surprising, therefore, that investigators at present are trying to determine whether factors in the environment are causally related to the occurrence of cancer in man. As has been true in the study of all diseases whose causes are unknown, the elements of the environment to which man is constantly or repeatedly exposed, e.g., the atmosphere, water, and foodstuffs, command the greatest share of attention.

Legitimate conjecture. Conjecture concerning possible cause and effect relationships is a common and sometimes an effective device in the development of new knowledge concerning disease. Many discussions concerning the possible relation of chemicals which may occur in foods to the occurrence of cancer in man have been held at scientific meetings and reports of these have recently appeared in scientific publications. Scientists involved in discussions of food additives and cancer recognize the *conjectures* as such, and ascribe importance to them only insofar as they may stimulate the kind of inquiries which will help advance knowledge. When the scientific discussions, either written or spoken, have been reported through the press and other news media for public information, however, the conjectural nature of the scientists' discussions has frequently been forgotten, misconstrued, or poorly stated. This has contributed to the present apprehension among consumers over the safety of the food supply, and to the concern among food manufacturers over the possible loss of consumer confidence.

In view of these circumstances, it is desirable that a statement be made clarifying for the public the present state of knowledge about the relation between food additives and occurrence of cancer in man.

Known facts. What is *known* concerning a definite relationship between ingestion of a substance and the subsequent development of cancer in man? Accidental repeated ingestion of "radium paint" or the use of so-called "radium water" has been followed by the development of cancer of the bone. The ingestion of certain aromatic amines, such as b-naphthylamine or 4-aminodiphenyl, through industrial exposure has been associated with the occurrence of cancer of the bladder. Epidemiologic evidence indicates that a prolonged intake of sufficient arsenic may result in development of cancer of the skin.

Do these materials occur in the food supply of the United States? Arsenic is the only one. It may occur in some foods in extremely small concentrations as a pesticide residue and is normally present in certain foodstuffs which have received no pesticide treatment. Insofar as is known, there is no danger from the amount of arsenic likely to be consumed from these sources under ordinary conditions.

If this were all of the story, probably no public apprehension would have arisen.

Cancer in experimental animals versus cancer in the human. Investigators studying cancer have induced the development of tumors in experimental animals by purposely exposing them to a number and

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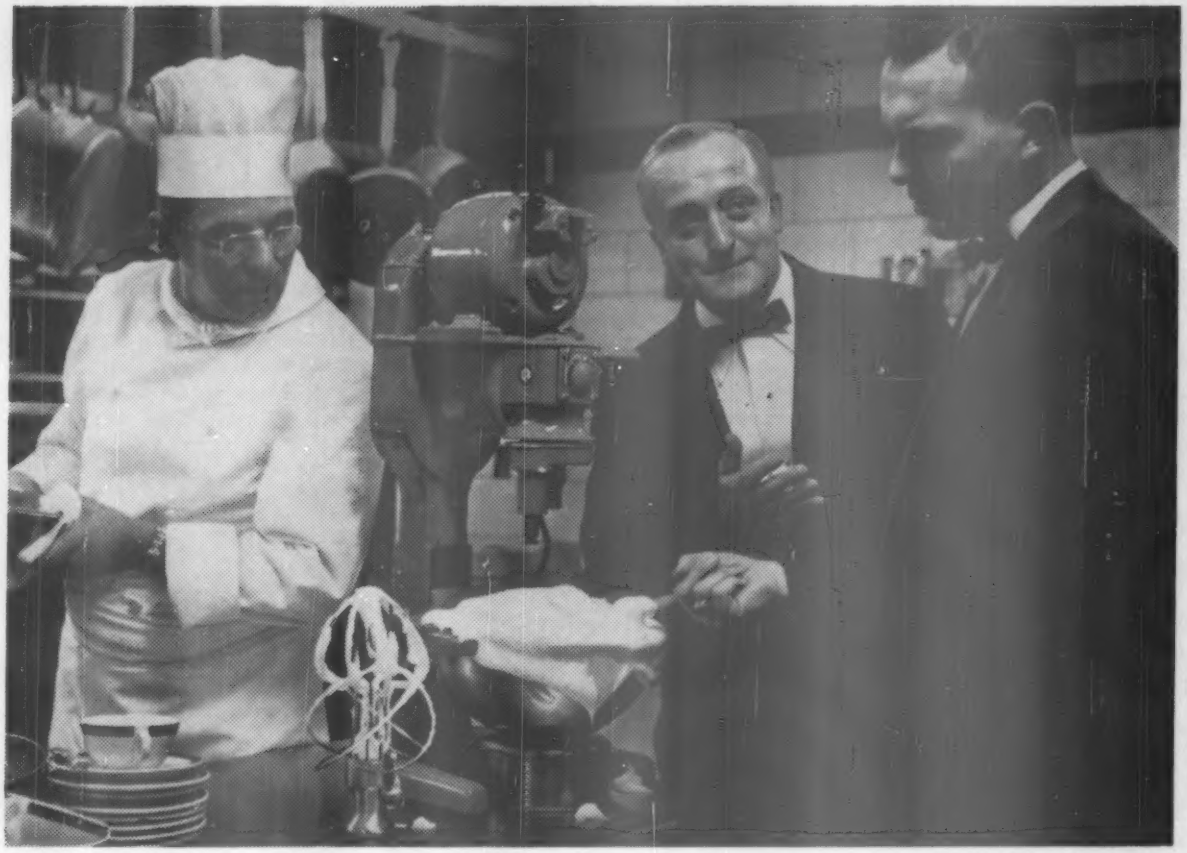

The mono man who doubles in vitamins

The man on the left is Joe. He's third cook at a famous hotel. Just finished working on a dinner for a meeting of pharmaceutical manufacturers. Now he's watching Bob Edwards (center), sales manager of our Oils Department, showing a fellow guest how a bit of DPi's distilled monoglycerides can make a lovely whipped-cream-like fluff out of homogenized milk. The fellow guest agrees that all this is interesting but suggests that Bob is at the wrong meeting. Bob stands his ground and points out that he's a monoglyceride salesman who doubles in vitamins.

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(Continued from page 8)

variety of chemicals. Such experimental cancer may be produced in animals by giving the agent by injection, by skin application, or orally. From this evidence and knowledge of how man may be like or different from the experimental animal in the metabolism, excretion, or storage of a particular chemical, the scientist can form an hypothesis as to how man might react to the ingestion of the chemical. In this respect, the conservative position would demand that substances that produce cancer in experimental animals should be excluded from human foods as a precautionary measure, even though it is known that a substance carcinogenic in one species is not necessarily carcinogenic in others.

Knowledge about possible cancer-causing agents in foods is, in general, at the point that studies are being devised and undertaken to test such possible relationships. This research is, by its very nature, expensive and time consuming. Years of study will be required to build definitive knowledge concerning all causes of cancer. There is a need to continue and expand present efforts to identify any relationships which may exist between environmental factors and the occurrence of cancer in man. Measures taken to safeguard the food supply can be only as effective as our state of knowledge permits. Government agencies, the food industry, and bodies such as the National Research Council are working together to facilitate the development of this knowledge and its effective application as soon as the information becomes available.

FEATURING THIS MONTH . . .

Disaster Feeding—a Dutch Approach[a]

Clarence Sterling[b]
University of California, Davis, California

WITH FULLY HALF ITS LAND SURFACE lower than the level of the sea at ordinary high tide, the Netherlands must continually maintain a vigil for the safety of its dikes and its sand dune barriers against the encroachment of the North Sea. Occasionally in its history, as in 1953, an extremely high tide may be combined with winds of very high velocity to permit the ocean waters to breach the carefully-tended barriers. (The 1953 floods drowned nearly 1800 people and left over 70,000 homeless.) Moreover, the Netherlands territory, being essentially an immense delta at the mouths of three rivers (Rhine, Meuse, and Scheldt), is also subject to flooding after heavy rains upstream or upon the melting of large masses of snow in the upland areas in the spring. Other emergencies have also been experienced. During the first world war, the interruption of normal commerce and of fishery operations brought about severe unemployment and food shortages. During World War II, the country was actually part of the battlefield.

Since disaster is always imminent in the Netherlands, as it is in every land, it is not without interest to inquire into the arrangements which have been made to face feeding problems in emergencies. The evolution of such arrangements will tend to reflect not only the country's economy but also its political history and its social customs.

In the United States, agriculture is a large-scale enterprise, is highly mechanized, and food surpluses are often produced. Preparation for feeding in disaster hinges mainly on the provision of food. Tremendous stores of food supplies are constantly maintained and are always available, even for large-scale feeding in other nations. (This has been remarkably demonstrated in recent years during emergencies in many parts of the world.) Research centers about problems of food quality, food storage, food packaging, proper foods for different environments, food acceptance, etc.

In the Netherlands, there is still a considerable number of workers in agriculture (about 20% of the total employed, in 1947), and there is much less mechanization in farming. (In 1954, 89% of Dutch farms were less than 50 acres in size, as compared with an *average* farm size of 215 acres in the U. S. in 1950.)[c] There is little real surplus in the food supply of the Netherlands. Yet it is interesting to note that here the major emphasis in disaster feeding seems to be not on the provision of ample stores of certain types of food but on the provision of a *hot meal* from available food supplies.

Dutch Approach Stresses Diet for Survival. By way of digression, the significance of this idea might be elaborated. There is a great difference between a diet which will permit survival and one which will enable a person to do continually heavy work and remain healthy. The Dutch approach appears to aim at the survival diet. It is proposed to take food from the normal channels of supply, thereby providing a minimum of 2000 calories per day per person, mostly in the form of carbohydrates. (Even without considering

[a] The author is deeply indebted to Drs. J. P. van Loon of the Netherlands Ministry of Agriculture, Fisheries and Food, for much of the factual material on disaster feeding plans and the use of the photographs that make up the figures for this article. However, the opinions stated are the author's alone and do not reflect those of the Royal Netherlands Government. The author is grateful to the USEF in the Netherlands for aid in support of his stay in the Netherlands via a Fulbright grant.

[b] In residence at the Laboratorium voor algemene en technische Biologie, Technische Hogeschool, Delft, Holland, during leave of absence from the University of California, Davis.

[c] *Landbouwcijfers.* 1955. Landbouw-economisch Instituut in samenwerken met Centraal Bureau voor de Statistiek. The Hague. *Agricultural Statistics.* 1955. U. S. Department of Agriculture, Washington, D. C.

(Continued on page 12)

IMITATION VANILLA BEAN FLAVOR BY FIRMENICH

Vanilla Bean Flavor by Firmenich stems from the vanilla bean. By original research, its components and desirable characteristics have been isolated, identified, and synthesized. By patient and painstaking laboratory techniques, they have been reconstructed for you in a precise and potent flavor material with all the aromatic qualities of the original. Vanilla Bean Flavor by Firmenich comes to you as an economical replacement of pure vanilla extract, of pure vanillin, or to use in combination in the manufacture of all products that you flavor with this most popular of flavors. Contains no coumarin.

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(Continued from page 10)

the existing import-export relationship in food materials, the country's agriculture furnishes roughly but 60% of the caloric requirements of the population.)[4] This is essentially the diet which was available during the less stringent years of the German occupation, 1940-1945.

Hot Meals from Central Kitchens. Although the amount of food may be meager, on the basis of the experience of the war years and of the earlier depression ("dole") years, it has been felt that a hot dish, of soup or cereal porridge, is best for sustaining morale. The provision of a hot meal implies the need for a kitchen, which is basically an energy source and a food container. Actually, during World War II, items such as "Veevoederketels" (cattle fodder kettles) were used in Rotterdam for feeding much of the populace following the heavy bombings in that city in 1940 (Figure 1). These were simply coal stoves plus large pots in which potatoes could be boiled for farm animals. However, such small units are regarded primarily as emergency measures. Much greater emphasis is being placed on the types of central kitchens which can be used to feed huge masses of people. So far as has been officially published, this is the main line of thinking of the Netherlands government on food for disaster feeding and on stress nutrition. Its main preparative efforts appear to be in this direction.

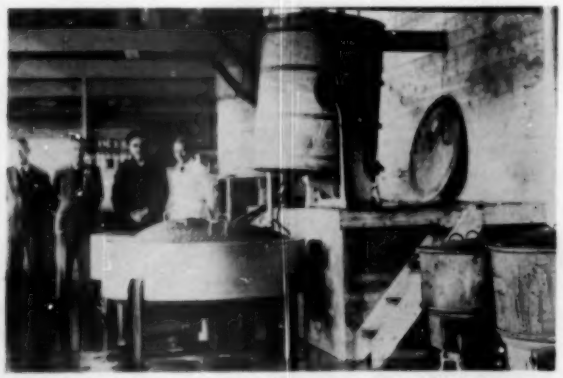

Figure 1. Typical mass-feeding kitchen facilities in Rotterdam in 1940. At extreme right are two "Veevoederketels." Two steam-injection pots for cooking potatoes and other vegetables stand upon the platform. These can be emptied by tilting, the contents being poured into the mixing bowls below, where the chef adds fat, spices, and other flavorings for soup.

Kitchen Facilities. In planned preparation for large-scale disaster feeding, several arrangements for kitchen facilities may be made. These various arrangements can be used in different combinations, depending on the extent of the emergency and upon their mutual availability.

First of all, if it be assumed that gas and electricity are usually not available in the disaster area, it is possible to have the food cooked elsewhere and sent in. During the war period, this was done often with "Gamellen," or double-walled containers (Figure 2). Such an approach is contingent on the availability of transportation facilities and energy sources not too far away.

Large kitchens, such as those of restaurants and schools, can be mobilized. For example, such a large kitchen prepares daily meals for some groups of school children of the city of Amsterdam. (Amsterdam already has had a wealth of experience with central kitchens. In the years of the first world war, 80,000 people were fed a hot meal a day in that city. During the later years of occupation, this experience proved invaluable in developing techniques and programs for feeding from large kitchens.)

Figure 2. Scene of "extra ration" for factory workers at midday meal. One of the double-walled "Gamellen" is shown in the foreground. (A usual early daily ration was 1 liter of soup. However, later in the war, this sometimes became ¾ or even ½ liter.)

Conversion of Food Processing Plants to Kitchens. Another approach, which is being pursued actively, is to arrange for the use of facilities in food processing industries (canneries, dairies, slaughterhouses, etc.) for cooking foods (Figures 3, 4). The idea here is to cook large masses of food quickly by injection of steam, under low pressure, into the food mass (Figure 5). Since the steam-generating facilities are already available in these plants, the problem becomes simply one of providing the fittings and containers for the injection-cooking technique. Discussions with industry are being carried on, and surveys are being made, with a view to using the basins and kettles already present in a factory; and plans are being developed to install additional, emergency equipment as money becomes available. In all parts of the country there are men in many factories who have had experience, during the war, in converting their factory into a gigantic kitchen. Factory managers will be trained to handle such conversions in emergency situations.

A related possibility to the above approach is also under consideration: to keep available in storage the pipes, fittings, wires, machinery and other types of apparatus which can readily be hooked up to existing

(Continued on page 14)

[4] *Dutch Agricultural Facts.* 1953. Netherlands Ministry of Agriculture, Fisheries and Food. The Hague.

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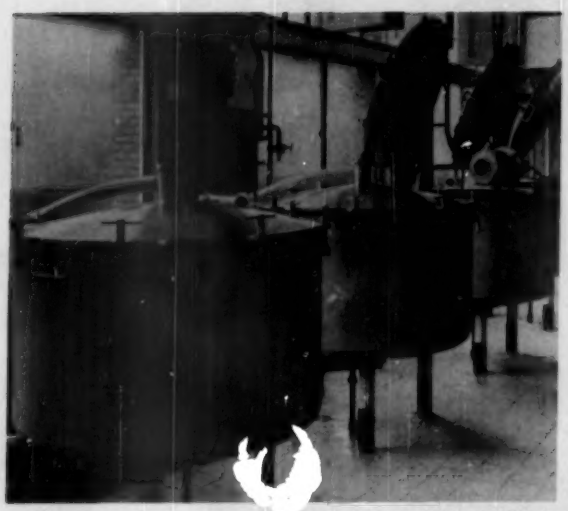

Figure 3. Low-pressure steam-injection cookers at Philips plant in Eindhoven (1944).

(Continued from page 12)

energy supplies. Much of this sort of construction, in sheds, provided cooking facilities in several towns during the first days after bombardment in wartime.

Lastly, "mobile kitchens"* have been developed. These consist of several trailer units, capable of being hauled by tractors, which together make a large, self-contained kitchen. The first assemblage of trailer vans was developed in 1941 (Figure 6), and there are now three such mobile kitchens in readiness. The kitchens are independent of outside energy sources initially but

Figure 4. Cooking facilities set up at Heineken's brewery in Amsterdam. Potatoes are being washed in vat in right foreground. Steam-injection cookers are at rear of room (1943).

dependent on outside food and water. (Although the water of the Netherlands is not very pure, it is very abundant.) There are separate vans, respectively, for generating steam and electricity (Figure 7), for purifying water and washing the utensils, for cooking—via the steam-injection method (Figures 8, 9), for meat preparation, and for vegetable washing and peeling (Figure 9). There is also an administration van, with sleeping quarters for the personnel. Such a mobile kitchen, constituting 8 vans in all, can prepare hot food for 40,000 separate servings (1 liter each) per day. At the present time, the kitchens are stored, together with other civil defense equipment, in a large garage in the neighborhood of The Hague.

By using all these kitchen arrangements together, it was possible in 1945 to feed daily hot meals to 2 million people, of a total population of 4.5 million, in the three provinces of North and South Holland and Utrecht. The total capacity available, from all the existing large kitchens (about 200) and all the convertible factories (about 1100) alone at that time was still greater: 10 million liters per day. (Note that the present population of the Netherlands is somewhat more than just 10 million.)

Figure 5. Preparing shredded vegetables for cooking children's ration during W. W. II. Such a picture could be taken at the present time: this kitchen, in The Hague, is still being used for school meals and for preparing stews, vegetables, and other courses for meals at official functions.

Aspects of Food and Fuel Supply. Despite the fact that the cooking arrangements seem to be so well in hand, it is well to stress that there is some danger in reliance on present food production in the event of a long-continued national emergency. Home production accounts for only 25% of the bread grains used and 10% of the oilcake. Likewise, animal feed is in deficit supply. Add to this the heavy density of population

(Continued on page 16)

Figure 6. Early mobile kitchen in 1941. The earliest mobile kitchen was a small trailer, carrying cooking facilities, being towed behind a small roadster.

* Dols, M. J. L., Loon, J. P. van, and Zoethout, H. De centrale keukens in de jaren 1940-1945. *Voeding* **7**: 67-75, 1946.
Loon, J. P. Van. Transportabele keukens. *Conserva* **3**: 262-267, 1955.

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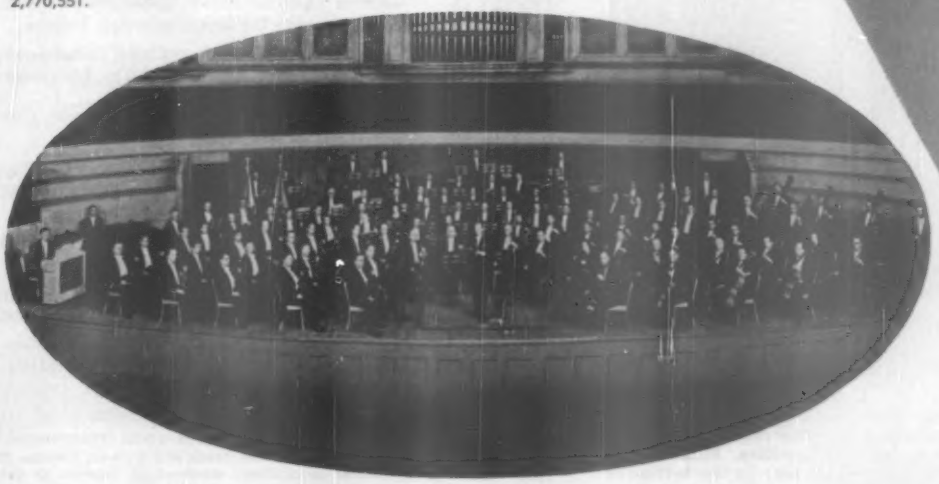

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(Continued from page 14)

(at 803 inhabitants per square mile,[f] the Netherlands have the greatest concentration in the world today), with a continuing pressure due to the highest birthrate in western Europe, and the food problem takes on a more serious aspect.

A similar situation is inherent in the fuel economy of the country. Whereas the production of coal has remained more or less constant between 1948 and 1954, the index of consumption of energy fuels has risen from 100 to 310. The great increase in fuel use is due to petroleum products, which are in short supply in the Netherlands—a deficit balance of 9.4 million metric tons

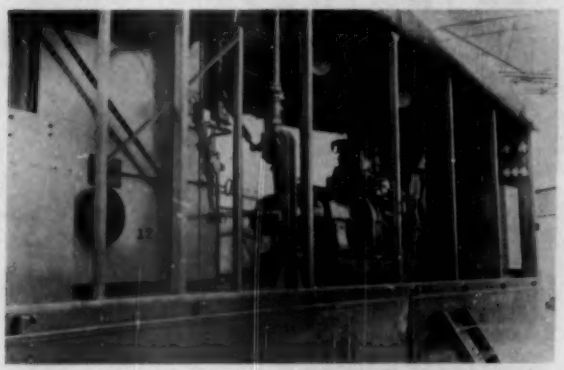

Figure 7. "Energy" trailer, showing Diesel-operated dynamo at 7 and steam boiler at 12. This is a unit of the modern mobile kitchen.

existing in 1954[f] (1 metric ton = 0.984 long ton). The dependence on imported petroleum fuels has been dramatically demonstrated by the restrictions on consumption attendant on the recent blockage of the Suez Canal. Consequently, any civil defense plans, for cooking and transporting foods, which require directly or indirectly combustion of imported petroleum products, deserve careful reconsideration.

[f] *Statistisch Zakboek;* 1955. Centraal Bureau voor de Statistiek. Uitgeversmij. W. de Haan N. V. Utrecht.

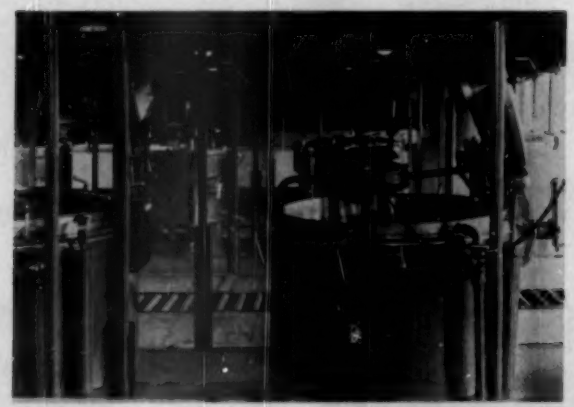

Figure 8. Interior of adjoining cooking vans in mobile kitchen. The cooking vans are in operating position, with the sides lowered to make a wider floor. The rollers between the vans permit easy removal of the filled Gamellen. Mixers (right foreground) mix the vegetables (and meat) in the kettles before cooking.

Figure 9. View of two cooking vans and two vegetable preparation vans of the operating modern mobile kitchen. The vegetable preparation vans are on each side of the cooking vans. Conveyors lift potatoes from the ground to the potato peelers at the forward ends of the vegetable preparation vans. (Not shown are the administration van and the vans for water-purification and Gamellen-washing, meat preparation, and steam and electricity generation.)

However, it should be noted that agricultural independence with respect to the grains is being slowly fostered in the Netherlands; while some of the fuel-burning equipment has been designed to be easily convertible to the use of coal, if necessary. It would appear then, to this observer, that the limiting factors in the present Netherlands disaster feeding program are more likely to be in the quantity and quality of food available and possibly in the energy supplies rather than in cooking capacity.

FUTURE MEETINGS FOR FOOD TECHNOLOGISTS

1957

February 12-14	Technical School for Pickle Manufacturers, Kellogg Center, Michigan State University, East Lansing, Michigan
February 15-17	Institute of American Poultry Industries 28th Fact Finding Conference, Municipal Auditorium, Kansas City, Missouri
February 16-19	Annual convention of the National Canners Association, Conrad Hilton Hotel, Chicago, Illinois
February 25	Indiana Canners and Fieldmen's Conference, Purdue Union Building, Lafayette, Indiana
February 26-27	Fifth Annual National Dairy Conference, Kellogg Center, Michigan State University, East Lansing, Michigan
March 15-16	Annual Food Technology Short Course, University of Missouri, Columbia
April 23-25	Annual Meeting of the Research and Development Associates, Food and Container Institute, Inc., Jefferson Hotel, Richmond, Virginia
April 30-May 2	A.O.C.S. Spring Meeting, Roosevelt Hotel, New Orleans
May 12-16	Seventeenth Annual Meeting of the Institute of Food Technologists, Penn-Sheraton Hotel, Pittsburgh, Pennsylvania
May 14-16	Second Annual Industrial Nuclear Technology Conference, Museum of Science and Industry, Chicago

An open invitation is extended to readers of FOOD TECHNOLOGY to send in to the Editorial Office, 11606 South Bell Avenue, Chicago 43, Illinois, notices of annual or national meetings of interest to food technologists.

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ANNUAL MEETING ANNOUNCEMENTS . . .

MEET THE PEOPLE PUTTING THE PITTSBURGH SHOW ON THE ROAD

THE COMMITTEES that man an annual meeting of the Institute of Food Technologists find their activities increasing in tempo and expanding with regard to details as each week brings closer the week of the meeting. The lot of the general chairman is not to be envied in the weeks preceding the Main Event. He is the man who must keep track of the sum total of pre-meeting problems and either know the man who has the answers or be the answer man himself. General Chairman of the Pittsburgh Meeting of IFT, and the man who will direct over-all proceedings May 12-16, 1957, is Dr. I. J. Hutchings, Manager of Food Research, H. J. Heinz Company. Co-Chairmen of the important program committee, Drs. R. E. Buck and C. R. Stumbo, were introduced in an earlier issue of FOOD TECHNOLOGY (September) along with the session chairmen. Upon the shoulders of these three men will rest the weighty responsibility of seeing that the Pittsburgh show goes on smoothly, punctually, and to the satisfaction of participants and attendees—who may number 2000.

Dr. I. J. Hutchings—General Chairman

To produce this desired result requires a division of labor. For example, it takes work to arrange for those attractive exhibits that beckon to food technologists who wish to know what is new, or improved, or available in instruments, machinery, flavorings, chemicals, packaging, and many other essentials to food manufacture and economy. Chairman of the exhibits committee for the Pittsburgh meeting May 12-16 is Charles J. Steinicke, Master Brewer, Duquesne Brewing Company of Pittsburgh. A master technician in the exhibits field and *ex-officio* a member of this committee is IFT Secretary, Colonel Charles S. Lawrence.

Highly important in keeping the show going—as a matter of fact in getting the show started—are the administrative committees. M. L. Crouthamel of H. J. Heinz Co. heads the vital finance committee. He and his committee members dispense the funds that pay for the facilities and services that make an annual meeting possible. Dr. Enio Feliciotti of Continental Can Company chairmans the registration committee. John K. McCarty handles banquets and special meals. K. F. Lang, H. J. Heinz Co., is in charge of arrangements for housing and session rooms. The scores of details that go into the management of a huge convention are kept track of and handled by the four committees just mentioned.

Dr. Enio Feliciotti — Chairman, Registration Committee

K. F. Lang—Chairman, Housing and Session Rooms Committee

"Public relations" is a term that today covers a multitude of activities. Perhaps its most apt applica-

(Continued on page 20)

Charles J. Steinicke—Chairman, Exhibits Committee

M. L. Crouthamel—Chairman, Finance Committee

F. C. Majorack—Chairman, Publicity Committee

F. J. Barry—Chairman, Printing Committee

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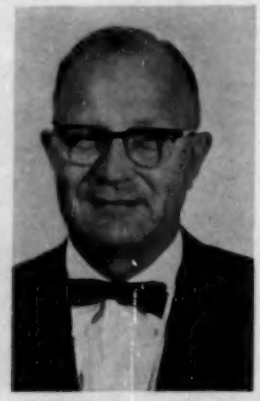
(Continued from page 18)

D. W. Leeper—Chairman, Courtesy and Information Committee

tion is to the duties involved in getting an annual meeting known to the membership and to the public. Chairman of the publicity committee is F. C. Majorack, H. J. Heinz Co. The printing committee is headed by F. J. Barry of the same firm. Courtesy and information, always a busy committee at an annual meeting, is headed by D. W. Leeper, also of H. J. Heinz Co.

Off-hour entertainment is another important division of this business of putting on an annual meeting. Chairman of the entertainment committee is George L. Reyman of the Stoner-Mudge Co. The chairman of the ladies entertainment committee, Helen B. Wigman, Mellon Institute, is introduced in the write-up on that commitee, which follows. V. W. Vaurio, U. S. Steel Corporation, is in charge of field trips—a phase of the meetings that can be as educational for food technologists attending the meetings as the papers or the exhibits. For young food technologists looking around the food world for opportunities these trips have special values.

George L. Reyman—Chairman, Entertainment Committee



V. W. Vaurio—Chairman, Field Trips Committee

David S. Nay—Chairman, Employment Committee

This point brings up the fact that an employment committee, headed by David S. Nay, Wm. J. Stange Company, has been set up to service the meetings. An annual meeting is an excellent market place for talent since industry and academic and government agencies are well represented there.

Although the chairmen of the Pittsburgh in '57 Meeting of IFT have been spotlighted in this write-up, it takes committee members as well as chairmen to perform the spadework that is required to put on a successful national meeting. In rounding off this presentation it is therefore appropriate to salute each and all of the committee members who will be putting the Pittsburgh show "on the road."

WOMEN'S ACTIVITIES AT 17th ANNUAL MEETING OF IFT

It will be three gala days for the ladies at the IFT Annual meeting in Pittsburgh next May. Miss Helen Wigman tells us that the Women's Entertainment Committee has just about completed plans for a program as varied as it is interesting. It includes everything from free breakfasts at a local food processing plant to swanky fashion displays at one of the nation's most elegant department stores. At the Cathedral of Learning, the women will have a peek at the old world when they visit the seventeen nationality rooms contributed by the many nationalities which have built Pittsburgh. On their visit to the University of Pittsburgh, the group will lunch at the Faculty Club and then witness the telecast of one of the city's most popular "live" shows.

Helen B. Wigman—Chairman, Ladies Entertainment Committee, Senior Fellow, Food Varieties Fellowship, Mellon Institute of Industrial Research.

The women will see in this place called Pittsburgh, the old and the new, a Pittsburgh that has been transformed from a smoky industrial center into one of the most beautiful cities in all America. That quiet English gentleman, Sir William Pitt, would be proud of the metropolis that celebrates his name.

And most important from a woman's viewpoint—along with a program that offers so many things to do—there will be lots of time for shopping and primping for the evening affairs. The committee promises the women that their visit to Pittsburgh will be a memorable one.

SYMPOSIUM ON METHODOLOGY OF SENSORY TESTING

The program for the annual meeting symposium, an innovation this year, is complete. The topics to be presented include two papers on the selection and use of taste panels and 6 additional papers, namely: consumer acceptance methodology, methods for rating quality and intensity of the psychological properties of foods, the flavor profile, the hedonic scale, a simplified uniform procedure for flavor difference evaluations, and a report on collaborative panel testing.

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The Nutrition Foundation Inc., New York, N. Y.

CONTRASTS IN NUTRITIONAL PROBLEMS in different parts of the world are striking. For example, in one group of countries there is a surplus of attractive foods that contain an abundance of all the nutrients required by man. We are fortunate to be in that group.

Dividends in terms of public health are increased greatly in this first group by steady advances in nutrition research, food technology, and education of the public. The life expectancy at birth is in the range of 70 years, and the most serious form of malnutrition is excess body weight—a result of continuously consuming an excess of calories. The impact of this wasteful, waistfull, practice is most striking in the upper age brackets. During the past two decades, for the first time in history, this country and others in comparable stages of development have reached a stage where food supplies are abundant, safe, of assured high nutritive quality, and available constantly to practically everyone. The specific nutritional diseases such as scurvy, rickets, pellagra, and goiter have practically disappeared.

Dr. C. G. King, Executive Director, The Nutrition Foundation, Inc. . . . stressed importance of world-wide attack on protein malnutrition at Symposium on Basic Nutrition Research held at Claremont Hotel, Berkeley, under joint auspices of the Foundation and Northern California Section.

There is still much progress to be made, however, in research, education and manufacturing, to accomplish the practical goals of optimum nutrition with greater convenience, economy and enjoyment. This trend introduces current concepts of health in terms of "preventive medicine." Better nutrition can still make notable contributions to public health by decreasing the incidence of obesity, irregular growth, tooth decay, anemias, fatigue, and many of the vague, chronic diseases. The research advances will be important, too, in guiding improved medical practices in the care of patients with metabolic diseases such as diabetes, arthritis, sprue, cystic fibrosis, celiac disease, liver failure, alcoholism, and heart disease.

The Other Half

Even with the resources that we have in this country, it will be noted that the last major deficiency diseases to be overcome were associated with a low intake of high quality protein foods. Pellagra, riboflavin deficiency, and the anemias associated with sprue were the latest to yield. The cereal food enrichment program made a valuable contribution to the gains; further advances resulted from improved education of low income groups, combined with a general rise in the quality of our food and in standards of living. A result was more uniform and increased consumption of milk, meat, fish, and eggs in the areas where severe deficiencies had been prevalent.

As a source of calories, however, these animal protein foods are often relatively costly. Hence, as the population density increases in areas where earning power is low, the economic pressure is toward reliance on foods obtained directly from plants. This trend may not involve a disaster, but it does imply a greater risk of protein malnutrition.

Severe forms of protein malnutrition such as kwashiorkor and pellagra generally reflect amino acid imbalances more than low intakes of total protein. This situation permits several approaches to the problem. The first and generally the preferred attack is based on increasing the consumption of animal protein foods. In practice, this usually means increasing local production, wider distribution to low income groups, and sustained education to reach the entire population. Adequate importation of milk, meat, fish, and eggs into areas characterized by protein malnutrition is not always possible, and this is especially true for countries whose economic resources are very limited.

A second major approach is through the use of legumes, cereals, and other plant sources of proteins that can be selected on the basis of a desired balance of available amino acids, even when no one or two of the products would be adequate. This gives the chemist an opportunity to work effectively with agriculturists, food processors, educators, and the medical profession.

A third approach is by adding synthetic amino acids in sufficient quantity to bring low-cost acceptable products to an approximately ideal content and balance, as measured in extensive tests with animals and human subjects. This approach is attractive and challenging to the chemical industry, but its extension to the human food supply is likely to be slow.

(Continued on page 30)

[a] Presented at meetings held jointly by the Nutrition Foundation with IFT Northern California Section, November 13, 1956, and at IFT Puget Sound Section, November 16, 1956.

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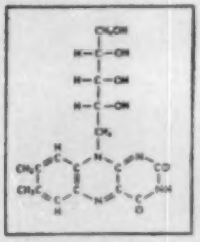
The Vital Story

A Quick History. Independent investigators, working separately to unlock several of nature's doors, sometimes open up unsuspected relationships. This happened with vitamin B_2.

Investigations. About 25 years ago, several groups, notably Warburg's, were investigating a "yellow enzyme" obtained from yeast. Almost simultaneously other investigators were studying a food factor that aided growth of laboratory animals.

What they found. Proceeding with chemical analysis of this growth factor, the team of Kuhn, György, and Wagner-Jauregg noted a relationship between the growth-producing agent and the "yellow enzyme." Their findings, and those of other researchers along similar lines, were published in 1933. Eventually, riboflavin and an essential part of the yellow enzyme were found to be identical and the unity of an essential nutrient and cellular metabolism was established.



Isolation of pure riboflavin was achieved by Kuhn and his co-workers, and by Ellinger and Koschara, in 1933.

Nomenclature. Known in the United States as riboflavin, this vitamin has also been called lactoflavin, ovoflavin, hepatoflavin, and vitamin G.

SYNTHESIS

By 1935, two eminent chemists, working separately, had synthesized riboflavin, practically in a dead heat. Prof. Paul Karrer of the University of Zurich, a collaborator of the Hoffmann-La Roche Laboratories, produced the first successful synthesis. Five weeks later Richard Kuhn of Germany announced his synthesis of the vitamin. Prof. Karrer subsequently shared the Nobel Prize in Chemistry for his work in vitamins and carotenoids.

The Karrer synthesis forms the basis for chemical processes in widespread use today by Hoffmann-La Roche and other leading manufacturers throughout the world. Riboflavin is also manufactured today by fermentation methods.

CHEMICAL AND PHYSICAL PROPERTIES

Riboflavin is yellow, slightly water-soluble with a greenish fluorescence and a bitter taste. Its empirical formula is $C_{17}H_{20}N_4O_6$. Vitamin B_2 produced by the Roche process is identical in every way with that occurring in nature.

How does vitamin B_2 work? Riboflavin is a vital part of nature's chain of reactions for utilization of carbohydrate energy. It has been found to be a constituent of many enzyme systems and is thus intimately connected with life processes. It is probably required by the metabolic processes of every animal and bird as well as by many fishes, insects and lower forms of life. (In certain animals, however, the requirement may be synthesized by bacteria within the intestine.)

In the cells riboflavin goes to work attached to a phosphate group. This substance, known as riboflavin-5′-phosphate or flavin mononucleotide, may in turn be attached to still another essential substance, adenylic acid, forming flavin adenine dinucleotide. Either nucleotide then is attached to protein, thereby forming an enzyme, and takes its part in oxidation-reduction reactions.

Requirements in Human Nutrition. As we have seen, vitamin B_2 is essential to life. We have no special storage organs in our bodies for this vitamin, although a certain level is maintained in various tissues, with relatively large amounts found in the liver and kidneys.

MEASURING METHODS

In the beginning, riboflavin activity was described in "Bourquin-Sherman units" and requirements were thought to be very small. Subsequent research showed otherwise. Milligrams of weight became the unit and the Food & Drug Administration of the U. S. Dept. of Health, Education & Welfare has established a minimum daily requirement of 2.0 mg. of riboflavin for all persons 12 or more years old. For infants it is 0.5 mg. These requirements are designed to prevent the occurrence of symptoms of riboflavin deficiency disease. The minimum daily requirement for this vitamin for children from 1 to 12 years has not been established by the F. & D. A.

Recommended allowances. The Food & Nutrition Board of the National Research Council has recommended the following daily dietary allowances of riboflavin, expressed as milligrams. These are designed to maintain good nutrition of healthy persons in the U. S. A.

			Boys	Girls
Men			1.6	
Women			1.4	
"	(3rd trimester of pregnancy)		2.0	
"	(Lactating)		2.5	
Infants,	1-3	months	0.4	
"	4-9	"	0.7	
"	10-12	"	0.9	
Children,	1-3	years	1.0	
"	4-6	"	1.2	
"	7-9	"	1.5	
Adolescents,	10-12	years	1.8	1.8
"	13-15	"	2.1	2.0
"	16-20	"	2.5	1.9

# of VITAMIN B_2 *by* Science Writer

(Riboflavin)

Deficiencies of vitamin B_2 appear in several ways in human beings. The eyes, the skin, the nerves, and the blood show the effects of too little riboflavin. Laboratory animals have demonstrated that a riboflavin-deficient diet can cause death of adults and can slow or stop growth in the young. Female animals, deprived of riboflavin in the diet, may produce offspring with congenital malformations.

Medical uses. To overcome and control deficiencies in human beings, physicians have pure riboflavin available for administration by injection or orally, by itself or with other "B" vitamins or multi-vitamin-mineral combinations.

How do we get our daily riboflavin? Vitamin B_2 has wide distribution throughout the entire animal and vegetable kingdoms. Good sources are milk and its products, eggs, meats, legumes, green leaves and buds. Whole-grain cereals have significant but not large amounts of riboflavin.

ADDITION TO FOODS

Cereal foods play a large part in our diet. To produce the white flour almost all of us want, millers are obliged to remove parts of the wheat that contain much of the grain's riboflavin and other nutrients. In addition, cereal grains are not rich sources of riboflavin. Millers meet this problem by *enriching* the grain foods for which federal standards exist with vitamins B_1, B_2, niacin and the mineral iron. In the case of vitamin B_2, however, they do more than *restore* the processed food to its natural riboflavin level; they *fortify* the food with enough of this essential vitamin to make it nutritionally more valuable than it was in nature.

Acting to protect the good health of millions of Americans, bakers and millers adopted *enrichment* of white bread and white flour in 1941. Since that time, other foods, such as macaroni products, corn meal and grits, farina, pastina and breakfast cereals have had their food value increased by enrichment with pure riboflavin and other vitamins and minerals.

When enriching, fortifying or restoring, food manufacturers add the necessary quantity of riboflavin (and other vitamins and minerals) to the food during processing, so that the finished product meets federal, state, and territorial requirements or contributes to the consumer an amount of the vitamin that dietary experts believe significantly useful.

PRODUCTION

Prof. Karrer's synthesis of riboflavin was a laboratory success. Adapting the process to commercial production, however, demanded original thinking by chemists at Hoffmann-La Roche. The production of riboflavin by chemical synthesis requires the production of ribose, a rare sugar, at an early stage in the process. This special sugar must be made inexpensively if the synthesis is to be practical. Sugar chemistry is a difficult matter. In a brilliant piece of work, the Roche chemical experts developed a method to produce ribose on a commercial scale by an electrolytic process, thus overcoming a most troublesome problem. Subsequently, Roche chemists developed the first practical synthesis for riboflavin-5′-phosphate, identical with natural flavin mononucleotide.

Picture three streams joining to form a river and you have a simplified idea of the Roche process for synthesizing vitamin B_2. O-xylene and glucose are processed separately to form xylidine and ribose respectively. These are joined to form ribitylxylidine, which is then converted to ribitylaminoxylidine. Starting separately with malonic ester, which is processed through intermediate stages to alloxan, the third "stream" is then joined with ribitylaminoxylidine to form riboflavin. Purification occurs at each step of the synthesis. Riboflavin 'Roche' equals or exceeds U. S. P. standards.

By the tons. So efficient is the Roche process that pure riboflavin is produced *by the tons* for use in pharmaceutical products and processed foods. An interesting development by Roche is the production of riboflavin in different forms related to the method of end use. 'Roche' Regular riboflavin U. S. P. is especially useful in dry enrichment premixes, powdered dietary supplements, pharmaceutical tablets and soft gelatin capsules. 'Roche' Solutions type is preferred for the manufacture of solutions having low concentration. 'Roche' Riboflavin-5′-Phosphate Sodium is a highly and rapidly soluble riboflavin compound favored for all pharmaceutical liquid products and some tablets, lozenges, and capsules. It has a more pleasant taste than the bitter U. S. P. riboflavin.

This article is published in the interests of pharmaceutical manufacturers, and of food processors who make their good foods better using pure riboflavin 'Roche.' Reprints of this and others in the series will be supplied on request without charge. Also available without cost is a brochure describing the enrichment or fortification of cereal grain products with essential vitamins and minerals. These articles and the brochure have been found most helpful as sources of accurate information in brief form. Teachers especially find them useful in education. Regardless of your occupation, feel free to write for them. Vitamin Division, Hoffmann-La Roche Inc., Nutley 10, New Jersey. In Canada: Hoffmann-La Roche Ltd., 286 St. Paul St., West; Montreal, Que.

(Continued from page 26)

Current emphasis upon health impairment by excessive intake of calories can easily lead us into the trap of forgetting that only within the past 15 years have we escaped from a national background in which nutritional deficiencies were persistent and widespread. Technically, we have always been in a position to produce enough protective foods to prevent deficiency diseases in the United States but poor education, low economic levels, occasional instances of alcoholism, a lack of basic information, limited transportation and less efficient food supplies than we have today, permitted severe deficiency conditions to continue through the centuries.

Protein Deficiency Unlikely in the U. S. A.

If the United States continues to advance economically and culturally, so that research, education, and food distribution can continue, we are not likely to encounter widespread health impairment as a result of protein deficiency within the next several decades or centuries. To be sure, the food industry must continue to be alert to the protein quality and total balance of all nutrients in our food supply. To do less would be unfair to the public and to industry. In this area of research and education, however, we should be careful to recognize the protein contribution that can be derived directly from plants, including bread and other cereal foods such as oats, rice, corn, rye, and macaroni. These foods, together with legumes, nuts, and vegetables, have important cultural and economic advantages when they furnish about one half of the protein intake. They share, also, with the animal-source foods, leafy vegetables and fruits in furnishing an abundance of all the energy, vitamins, and minerals essential for normal human health.

Some day we may reach a stage that makes it physiologically and economically advantageous to use synthetic amino acids or synthetic peptides and proteins as major supplements to the available food supply, but such a period does not seem to be close at hand. We shall need much more information than we now have before most nutrition scientists would be willing to endorse the practice. In animal foods, there is substantial and encouraging progress in this direction already and the trend is likely to continue. Methionine is well established. Lysine is fairly promising except for cost, and still others are on the horizon. Preliminary estimates of total sales in 1955 indicated about two million pounds at a value of about $4,800,000.

A similar situation exists with respect to such foods as cultured algae, yeasts (beyond their use in breads), fungi, plankton, and bacteria. Their utilization in animal foods has reached important levels already and probably will grow steadily with advances in technology and nutrition research but their introduction into human food supplies, as major items to compete with roast beef, milk, cheese, salmon steaks, eggs, and bread, is not likely to follow rapidly in the immediate future.

Meanwhile, a higher degree of efficiency and scientific study in the fisheries industry appears to offer one of the greatest opportunities for substantial and permanent advance. F. J. Weiss has given a valuable picture of prospects in this respect.

Risks associated with long-term effects of imbalances, the use of racemates instead of natural isomers, and the effects of marked differences from proteins in the time sequence of absorption from the intestinal tract have not been explored adequately in human nutrition. The evidence from experiments with animals, as reported by C. A. Elvehjem and other careful investigators, shows that risks to health in this respect cannot be taken lightly by either the chemical industry or the food industry.

In areas where the quality of protein intake is handicapped by inadequate consumption of animal protein, the imbalances are quite varied. Low intakes of methionine, tryptophan, lysine, isoleucine, and threonine are often at fault, and excess intakes of leucine and glycine can cause injury when the intakes of isoleucine and tryptophan are low. Among other recent papers in this field are those by R. R. Williams, R. Block and W. H. Cole, with special reference to the probems of cost and practical outlook for the future. W. B. Bradley, C. H. Harrel, and others have pointed out the economic and nutritional advantages furnished by modern breads that contain non-fat milk solids.

Human populations where protein malnutrition is widespread cannot aflord to use products so expensive as the presently available synthetic amino acids. The economic situation makes it necessary to seek answers primarily by producing and consuming increased quantities of animal protein foods and by supplementing these products with plant protein foods that have reliable nutritive value. In both of these directions, progress can be made rapidly. There is no doubt of the great need for giving strong support to measures of this kind, pending further progress in the chemical industry and in human nutrition research.

In 1955 we reached a per capita protein consumption of 98 g. per day, of which two-thirds was from animal sources. This is a favorable picture when one considers that the liberal Recommended Dietary Allowances of the National Research Council suggests 55 to 100 g. per day for adults, of which 50% should be from animal sources.

Protein Malnutrition, a Problem in Other Lands

In many parts of the world the picture is very different from ours. It is grim indeed where protein malnutrition is regarded as a dominant factor in causing sickness and death. Standard practice for millions of people is still based upon weaning youngsters onto a diet consisting almost entirely of foods high in starch such as rice, corn, millet or cassava, supplemented by variable intakes of a legume such as beans or peas. Tragically, these combinations often do not meet the human infant's requirement. The result in large areas of Central and South America, the Near East, Africa, the Far East and India is a rapid onset of malnutrition after weaning, with resultant stunting of growth, injury to the glandular organs, disturbances of the central nervous system, a high incidence of intestinal infections and

(Continued on page 47 following technical papers)

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SELECTED ABSTRACTS . . .

Compiled by H. A. Campbell

ANALYTICAL METHODS

Volatile compounds produced by apples. I. Aldehydes and ketones.

MEIGH, D. F. (Ditton Lab., Larkfield, Maidstone, Kent, Engl.). *J. Sci. Food Agr., 7,* 396-410 (1956).

Methods were developed for analyzing the volatile aldehydes and ketones present in the air surrounding gas-stored apples. These methods were used to compare the compds. produced by three varieties of apple, one susceptible and the other two resistant to superficial scald. The comparison did not reveal any fundamental differences between them. Of the aldehydes and ketones produced by the three varieties, Me_2CO predominated.

BIOLOGICAL SCIENCES

BIOCHEMISTRY

Enzyme alteration by ionizing radiation.

ARONSON, D. L., FRASER, M. J., AND SMITH, C. L. (Univ. of Cambridge, England). *Radiation Research, 5,* 225-37 (1956).

The increase of intracellular yeast catalase activity (Euler effect) induced by exposure of the cells to X- and γ-radiation was shown to be due to a change in the phys. state of the enzyme (enzyme alteration). Calculation of the target size for the alteration process yields a value corresponding to a substance of mol. wt. 2×10^6. This might be ribonucleic acid which is known to be intimately assocd. with yeast catalase. Calculations from the dose of X-radiation needed to inactivate fully altered yeast catalase lead to a value of 250,000 for the mol. wt. of that enzyme. The results show how ionizing radiation can affect the enzymatic activity of the more radiosensitive portions of the cell. This leads to a disturbance of the physiol. balance of the cell before appreciable direct inactivation of the enzyme itself occurs.

The action of ultraviolet radiation on yeast catalase.

KAPLAN, J. G., AND PAIK, W. K. (Dalhousie Univ., Halifax, Canada). *J. Gen. Physiol., 40,* 147-69 (1956).

The effect of prolonged UV irradiation (mostly 2537 A) on the catalase activity of an aq. yeast suspension was divisible into 4 periods. First, the period during which the cells lost their ability to form colonies, but during which no change in catalase activity was noted. Second, the period during which a considerable rise in catalase activity (Euler effect) occurred. Third, a rather long period during which irradiation led to no diminution in the catalase activity of the maximally active suspension. Fourth, the period of photoinactivation of the intracellular enzyme, which was quite similar to that of the cryst. enzyme *in vitro.*

Proteolytic enzymes in human and cow's milk.

STORRS, A. B., AND HULL, M. E. (Armour and Co., Chicago). *J. Dairy Sci., 39,* 1097-1103 (1956).

Human milk contains proteolytic enzymes. About one-third of the proteolytic enzymes added to cow's milk in the processing of enzyme-treated milk survive pasteurization. Human milk and pasteurized enzyme-treated cow's milk contain substantially equal amts. of proteolytic enzymes. Raw or pasteurized cow's milk contains about one-fifth as much proteolytic enzyme as either raw human milk or pasteurized enzyme-treated milk. These results provide a new concept in the nutrition of infants as well as others who do not tolerate cow's milk.

MICROBIOLOGY

A source of coliforms in frozen concentrated orange juice. Fruit surface contamination.

WOLFORD, E. R. (Western Utilization Research Branch, U.S.D.A., Pasadena, Calif.). *Appl. Microbiol., 4,* 250-3 (1956).

Coliforms were found on surfaces of fruit sampled from groves, although *Aerobacter* organisms were isolated in most cases. *Escherichia coli* was found on one fruit sample, and evidence pointed to use of org. manure fertilizer around the tree as the possible source of contamination. Incidence of coliforms was more frequent on fruit which had been subjected to bruising during bulk handling. The presence of coliforms on processing-grade fruit as it arrived at products plants indicated that the presence of organisms in juice produced from this fruit is not positive evidence of unsanitary conditions in the plants.

Penetration and growth of Salmonella in shell eggs.

STOKES, J. L., OSBORNE, W. W., AND BAYNE, H. G. (Western Utilization Research Branch, Albany 10, Calif.). *Food Research, 21,* 510-18 (1956).

Penetration and growth occurred just as readily with the non-motile strains as with the motile strains. The course of infection consists of a period of relatively slow penetration of the shell membranes during the first wk. of incubation, followed by slow bacterial multiplication within the egg during the second wk. and finally by rapid growth to very large nos. during the third wk. The data suggest that shell eggs which must be stored for several wks. or longer should be kept below 10° C. to prevent possible infection with *Salmonella.*

NUTRITION

Amino acid imbalance as related to methionine, isoleucine, threonine and tryptophan requirement of the rat or mouse.

SAUBERLICH, H. E. (Alabama Polytechnic Inst., Auburn). *J. Nutr., 59,* 353-69 (1956).

The results suggest that the imbalance effect may be a general phenomenon assocd. with specified conditions for probably most of the essential amino acids and possibly even for some of the "nonessential" amino acids. Results of the present study emphasize again that the amino acid requirements of the rat are not a const. factor but are related to the diet employed and in particular to the protein or N level of the diet. In order for efficient and max. tissue protein synthesis to proceed in an animal, the diet must furnish amino acids not only in sufficient quantity, but also in proper balance.

Effect of added lysine on growth of rats fed a cereal and milk diet.

SARETT, H. P. (Mead Johnson Research Labs., Evansville, Indiana). *J. Nutr., 60,* 129-35 (1956).

The gain in wt. of male weanling rats on a diet of 70% cereal and 30% powdered whole milk was not significantly increased by the addn. to the diet of 0.14 or 0.28% of L-lysine (equivalent to 0.2 or 0.4% of L-lysine added to the cereal) nor by addn. of a supplementary vitamin mixt. The diet of 70% cereal and 30% powdered milk contained approx. 1.08% lysine, which exceeds the requirement for growth, 1% found by Rose.

Reproduction of rats fed protein-free amino acid rations.

SCHULTZE, M. O. (Univ. of Minnesota, Inst. of Agr., St. Paul). *J. Nutr., 60,* 35-45 (1956).

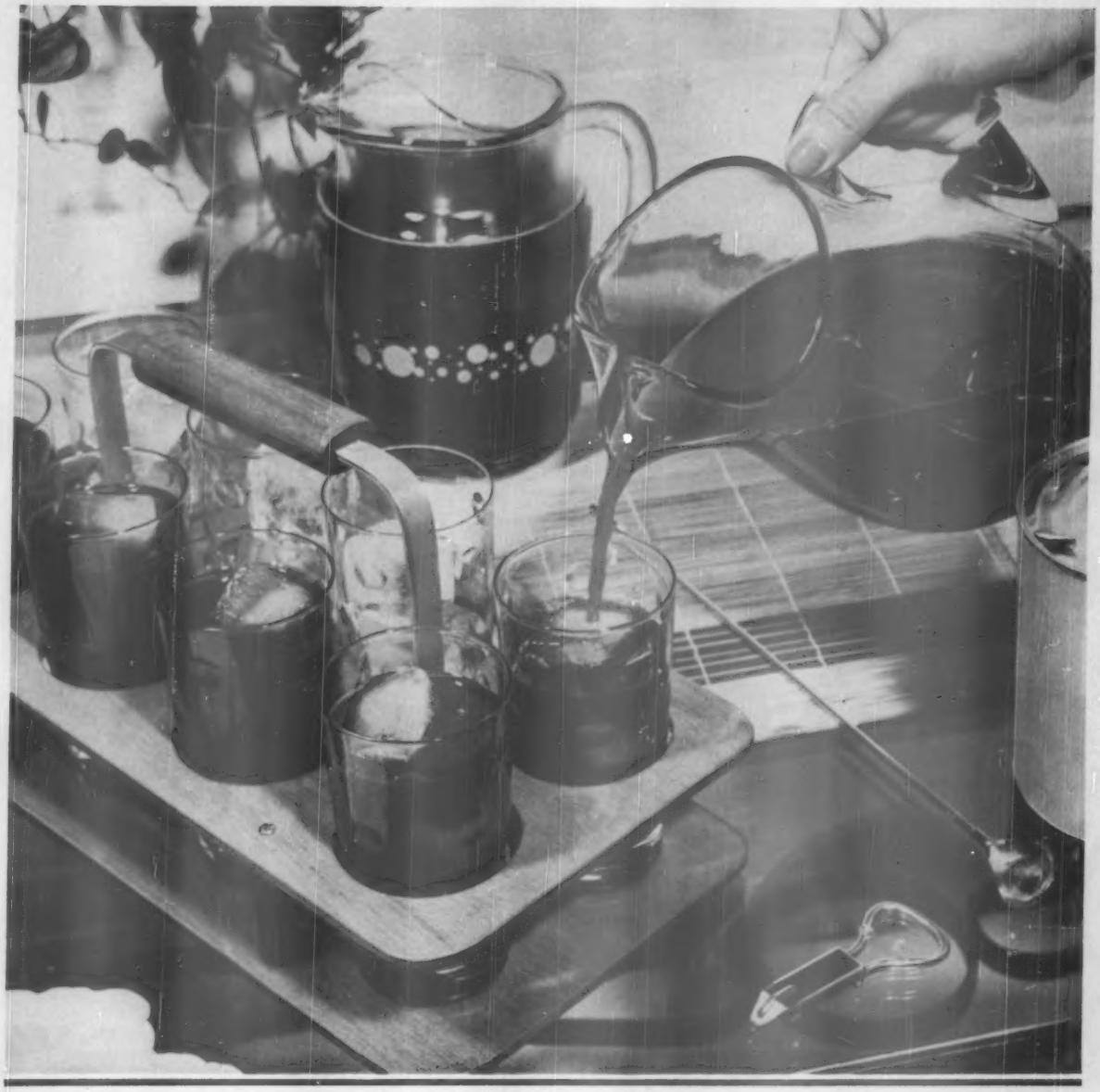
Protein-free rations contg. mixts. of 10, 11 or 16 amino acids were fed to rats during growth, two pregnancies, and lactation, in one instance for as long as 4 successive filial generations without evidence of gradual deterioration of the reproductive or lactation performance. The nutritive value of a protein-free ration is affected by the relative proportions in which the individual amino acids are present as well as by the abs. amts. None of the rations used was completely adequate for optimum preweaning or postweaning wt. gains of the young or for the prevention of fatty livers during lactation.

Development of fatty livers during lactation of rats fed amino acid rations.

HALLANGER, L. E., AND SCHULTZE, M. O. (Univ. of Minnesota, Inst. of Agr., St. Paul). *J. Nutr., 60,* 25-33 (1956).

Rats fed protein-free amino acid rations developed hypertrophied and severely fatty livers under the stress of lactation. After lactation the concn. of liver lipids decreased. When lactation of rats was supported by an adequate diet some hypertrophy of the liver was observed but the lipid content of the liver decreased.

(Continued on page 34)



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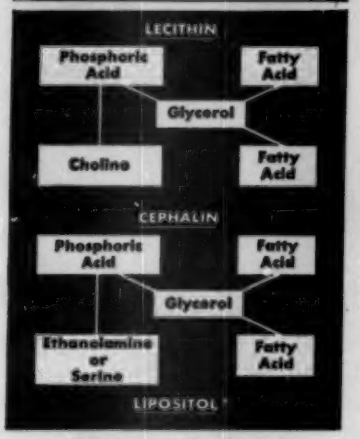

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(Continued from page 32)

The effect of radiation sterilization on the nutritive value of foods. I. Biological value of milk and beef proteins.

METTA, V. C., AND JOHNSON, B. C. (Univ. of Illinois, Urbana). *J. Nutr., 59,* 479-90 (1956).

The effect of irradiation sterilization and of heat sterilization on the nutritive value of milk proteins and beef proteins were studied by the Thomas-Mitchell method. The irradiated beef when incorporated into a balanced diet was found completely acceptable to the rat while the diet contg. irradiated milk was consumed reluctantly. Irradiation sterilization did not affect the apparent or true digestibility of the beef (100%) or of the milk protein (98%). The biol. value of the beef protein (78%) also was not affected upon irradiation but the biol. value of the milk proteins (90%) was reduced by 8% upon irradiation as compared to a reduction of 6% due to heat sterilization. The possibility of irradiation damage to the S amino acids is discussed.

Optimum ratio of saturated to monounsaturated fatty acids in rat diets.

HOPKINS, C. Y., MURRAY, T. K., AND CAMPBELL, J. A. (National Research Council, Ottawa, Canada). *Can. J. Biochem. & Physiol., 33,* 1047-54 (1955).

Fat mixts. were prepd. in which the content of linoleic acid was held const. at approx. 10% of the total fatty acids, but the ratio of satd. to mono-unsatd. acids was varied from 3.5:1 to 1:8. These mixts. were added to a low-fat fox chow diet at the rate of 20 parts fat to 80 parts fox chow, and the resulting mixts. were fed to weanling rats in one expt. and to semiadult rats in another. The feeding period was nine wks. In both expts. the male rats showed significant differences in final wt. between the diets. Best wt. gains were made on the fat mixt. in which the fatty acid ratio was close to that of normal rat depot fat, viz. one part of satd. acid to two parts of mono-unsatd. acid. Female rats did not show significant differences in wt. gain on the various fat mixts. Excretion of lipid in the feces showed a tendency to increase with increasing ratios of unsatd. to satd. fatty acids in the diet.

A comparison of the biological values of dietary protein incorporated in high- and low-fat diets.

METTA, V. C., AND MITCHELL, H. H. (Univ. of Illinois, Urbana). *J. Nutr., 59,* 501-13 (1956).

The effect of fat as compared to isocaloric amts. of carbohydrate on N utilization in the severely protein-depleted and the growing rat was investigated using the Thomas-Mitchell procedure. The main difference between the two groups of test diets was in the fat intake. When fed in isocaloric amts., dietary fat and carbohydrate have essentially the same effect on protein utilization in both the protein-depleted rat and the growing rat. Neither the apparent digestibility nor the true digestibility of casein is improved by the higher dietary fat level.

The biological value of oils and fats. IV. The rate of intestinal absorption.

THOMASSON, H. J. (Unilever Research Lab., Zwijndrecht, Netherlands). *J. Nutr., 59,* 343-52 (1956).

The rates of absorption of 18 natural oils and fats of vegetable and animal origin were studied. These oils and fats could be divided into 5 groups according to a decreasing rate of absorption: butterfat, maize oil, cottonseed oil, beef tallow, coconut fat, soybean oil, sunflower oil, groundnut oil and olive oil; sesame oil, lard, palm fat and whale oil; shea butter and herring oil; rapeseed oil, poppyseed oil and kapokseed oil. A significant correlation was shown to exist between rate of absorption and growth action of the oils and fats.

Fat deposition in coronary arteries of rats exposed to cold.

SELLER, E. A., AND YOU, R. W. *Brit. Med. J., 1,* 815 (1956); *Nutrition Revs., 14,* 284-5 (1956).

Investigators working with Wistar rats noted stainable lipids in coronary arteries of an exptl. group kept in a cold room (1-3° C.) for 10 to 18 mons. and none in a control group. After 12 to 13 mons. exposure to cold also resulted in a moderate increase in total blood lipids and free and bound cholesterol. The role of choline in the pathogenesis of cold induced lesions was also studied using a hypolysotropic ration with and without choline supplementation. The rats housed in the cold, paradoxically showed a high incidence of fat deposition in their coronary arteries only when fed the diet supplemented with choline.

Lard vs. a vegetable fat in relation to liver and serum cholesterol.

OKEY, R., AND STONE, M. M. (Univ. of California, Berkeley). *J. Am. Dietet. Assoc., 32,* 807-9 (1956).

Two studies were reported, in which matched groups of rats were fed (a) lard and (b) vegetable fat, each with and without cholesterol. The lard fed male rats tended to store somewhat more liver cholesterol than did those fed equal percentages of vegetable fat. Differences were lessened by an increase in the protein content of the diet. Lard-fed females showed smaller differences in liver cholesterol than did males. Serum cholesterols were, however, consistently higher in females.

Studies in calcium metabolism. Effect of food phytates on calcium ^{45} uptake in boys on a moderate calcium breakfast.

BRONNER, F. (Massachusettes Institute of Technology). *J. Nutr., 59,* 393-406 (1956).

Phytates do not exert a significant effect on Ca^{45} absorption when the meal provides 239 mg. of Ca and when the phytic P intake is 80 mg. Because this ratio of Ca to phytic P is typical of diets in the United States, it may be concluded that food phytates are of no nutritional concern in this country.

Metabolism and requirements of iron in the human.

MOORE, C. V., AND DUBACH, R. (Wash-

(Continued on page 36)

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(Continued from page 34)

ington Univ. Sch. of Med.). *J. Am. Med. Assoc., 162,* 197-204 (1956).

A discussion, whereby it is pointed out that the recommendations of the Food and Nutrition Board for Fe are adequate; however they do not provide a wide margin of safety.

Iodine and fluorine in nutrition.

Nature, 177, 912-14 (1956).

The daily intake of I for man has been put at 100-150 μgm.; only about 10% of this is usually supplied by the H_2O, even in areas free from goiter, so the remainder must be obtained from food sources. So far, it has not been possible to prove that F is an essential dietary element; although it is now generally accepted that the use of H_2O supplies with 1 or more p.p.m. of F is assocd. with a lower incidence of dental caries than waters with less.

Influence of food and energy restriction and subsequent recovery on body composition and food utilization of rats.

MEYER, J. H., LUEKER, C. E., AND SMITH, J. D. (Univ. of California, Davis). *J. Nutr., 60,* 121-7 (1956).

Growing rats were restricted in their food or energy intake for 3 or 4 wks. at 70% of the food intake per unit of body wt. of the ad libitum-fed controls. They were subsequently fed a total amt. of food equivalent to that consumed by the controls. Even though there was little difference in wt. gains between rats restricted either by energy or total food intake, differences were apparent in body compn. A greater proportion of fat was found in the bodies of the rats which were restricted in total food intake and subsequently fed adequately.

Appetite and obesity.

MAYER, J. *Sci. American, 195,* 108-16 (1956).

The author describes physiol. expts. which are attempting to solve the complexities of the mechanism regulating appetite. The causes of obesity are traced to three sources: heredity, injury and unfavorable external factors relating to nutrition and exercise.

Diet and anemia: zymotic and other factors.

PLATT, B. S. (Med Research Council Labs., Holly Hill, London). *Proc. Nutrition Soc., 15,* 103-10 (1956).

There is much evidence that an adequate supply of protein is necessary for normal haematopoiesis, but the idea that there is a prior claim for protein in the formation of new red cells has tended to draw attention away from protein deficiency as an important cause of anemia. Anemia that is assocd. with infection, trauma and neoplasia might well be a manifestation of a diminished supply of protein available for haematopoiesis.

Life span and causes of death in inbred mice in relation to diet.

CHIUNG PUH YEE, Y., VISSCHER, M. B., AND KING, J. T. (Univ. of Minnesota, Med Sch., Minneapolis). *J. Gerontol., 11,* 364-71 (1956).

(Continued on page 39)

Ultra-critical temperature problem solved by Chicago Stainless Equipment Corp.

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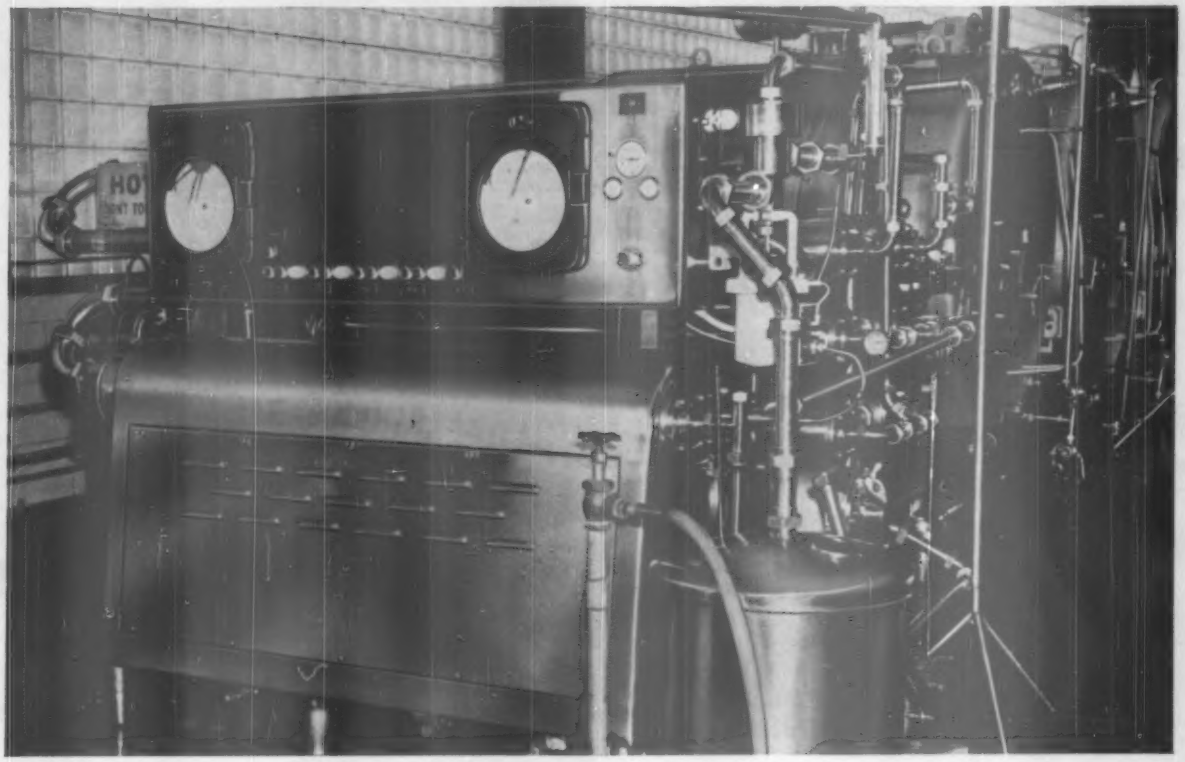

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The "COF Effect" in Frozen Citrus Products[a]

J. S. Blair, Edith M. Godar, H. G. Reinke, and J. R. Marshall
Research and Technical Department, American Can Company, Barrington, Illinois

(Manuscript received June 13, 1956)

IN THE COURSE OF THE REMARKABLE EXPANSION of the frozen citrus industry within the last few years, a peculiar flavor effect has been encountered for which no simple and easy explanation offers itself. The effect occurs in only a small proportion of the frozen pack; yet it has occasioned inconvenience, uneasiness, and a certain amount of monetary loss to the industry. Consequently, a better understanding of the causative factors is much to be desired. The present paper reports the results, largely qualitative, of a considerable amount of exploratory laboratory work on this problem. This work has afforded part of the evidence which we wish to cite in support of the suggestion that the fundamental factors causative of the phenomenon are natural physiological processes in the fruit itself; processes whose significance is not specifically restricted to citrus products. The suggestion is that these processes are those which are connected with the biosynthesis of lipids. However, a very important part of the evidence, which must be considered, consists of field observations made by technologists of the citrus industry. The help of these other workers in studying this problem is gratefully acknowledged.

Description of the phenomenon. The adjectives used to describe the sensory perception of this effect are, "castor-oil," "tallowy," and "cardboard." The term "castor-oil" refers primarily to the detection of certain odorous components which remind observers of flavor effects observed in prior experience when castor oil was administered mixed with orange juice in an effort (obviously not entirely successful) to disguise the flavor or odor of the oil. The term "tallowy" seems to have reference to a "mouth-feel" type of experience. Some persons have difficulty in perceiving the odor and appear to rely largely upon the "mouth-feel" perception in detecting the effect, whereas other persons who are extremely sensitive to the odor appear to attach little importance to the perception of tallowiness. Average observers, however, seem to be able to sense both of these effects if they are intense enough. The evidence seems to justify the thought that the two effects are ascribable to distinct but correlated phenomena. The origin of the phrase, "cardboard off-flavor" is more obscure but may have reference to a sensitory resemblance to that effect in milk products which has come to be referred to as "cardboard" as well as "oxidized." Finally, some observers refer to the effect under discussion as a "citrus-oxidized flavor," and there is evidence that redox level sometimes can be a limiting factor in its development, although evidence will be presented herein to show why we think that the mechanisms of the citrus effect and of the milk effect must be different. The term "COF effect" is offered as a phrase to suggest all these aspects of a complex situation without commitments as to particular sense-impressions or particular assumed modes of causation.

Observations which receive general concurrence:

1. COF may be encountered in the frozen concentrated juice of both orange and grapefruit, and in frozen grapefruit sections.

2. When COF appears, it does so only after a period of frozen storage which may vary from about two days to several weeks.

3. However, in more prolonged storage the effect tends to disappear. The disappearance is slower at 0° F. than at higher refrigeration temperatures. The odor disappears first; the tallowiness persists much longer and sometimes for a year or more.

4. A very striking fact is the extreme package-to-package variation which may occur. Early-orange concentrates are sometimes packed and frozen in 55-gallon drums which are held for later blending with other citrus materials. Drums filled from the same evaporator batch will show extreme variance in intensity of apparent COF at any one examination. The same variance sometimes manifests itself even in six-ounce cans of grapefruit concentrate packed in sequence through the equipment and packed in the same case. Careful observers have even remarked that there seem to be definite differences in COF intensity between grapefruit sections packed and frozen in the same package. One may say that in this problem one cannot count on obtaining reliable replicates, and investigation by means of experimental packs is correspondingly difficult. It seems possible that this variance may be due, in part at least, to unexplained differences from sample to sample in the rate of appearance and disappearance of the effect.

5. This appearance and disappearance, and the prevalence of borderline cases, is an indication that COF is allied to normal flavor factors and may well consist of normal flavor components which are present in abnormal relative concentrations.

6. There is very strong evidence that COF is correlated with physiological immaturity in the fruit. Here it may be necessary to distinguish between two concepts. It has become customary in the citrus industry to use the term "maturity" to represent a sugar/acid ratio which confers flavor acceptability. The word "ripeness" could be used to designate this quality and the term "maturity" would then describe that stage of development at which certain enzyme systems become relatively dormant. It has been very generally observed that early varieties of orange such as Hamlin are quite prone to develop COF, whereas it is unusual for late varieties such as Valencia to develop the defect in marked degree. Hamlins require only about 10 months

[a] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Mo., June 13, 1956.

from blossom to harvest while Valencias may require 16 months; this indicates that Hamlins may always have the more dynamic enzyme systems at corresponding stages of ripeness. It has also been observed that Florida grapefruit harvested in early February, for instance, is much more prone to develop COF than is grapefruit harvested a month later. Yet the February grapefruit may have a quite acceptable sugar/acid ratio and when canned, rather than frozen, the product may be of high quality.

7. The nature of the COF-malodor is of a type which suggests some kind of relationship to the lipid constituents of the product, though it is hardly of a character to enable one to speak of oxidative rancidity with any assurance. It is rather, almost by definition, suggestive of the odoriferous minor constituents which are naturally present in unrefined vegetable oils and which to some extent differentiate one such oil from another. The COF-malodor is volatile. This is shown, not only by laboratory work such as that described herein, but technologists in charge of quality control in the field have stated that when the effect has developed in drums of "Early Orange Add-back" as mentioned above, the material may be passed briefly through the evaporator again, with resultant benefit to the flavor of the ultimate blended product. This is in accord with other evidence which shows that when COF has once run its course, it does not recur in the same citrus material.

8. COF is encountered most frequently in products which are relatively bland in odor, and is not easily perceived when the aromatic natural components of citrus are present in relatively high concentration. Such aromatic components may be those derived from the essential (peel) oils of orange or grapefruit, or they may be pulp constituents in a fragrant type of orange such as the Pineapple variety. Evidence will be presented herein to show that this effect is at least partially one of mere odor "masking," though some chemical interaction of odor components remains possible. Opinions vary as to whether such "masking" expedients are completely successful, but they certainly do mitigate the severity of the COF effect. Then, too, many persons think that, regardless of COF, consumer opinion is veering in the direction of better acceptance for frozen products, higher in natural flavor components.

Role of redox level. There are two lines of evidence that point to a correlation of COF with redox level. Dr. F. W. Wenzel and his colleagues at the Florida Citrus Experiment Station and Florida Citrus Commission Laboratory have described to the present authors their extensive organoleptic evidence to show that when citrus materials are passed briefly through a Waring blender before being put into frozen storage, the intensity of COF is often greatly enhanced, and in some cases to such a degree as to constitute the difference between organoleptically-perceived "presence" and "absence" of the effect. However, these workers have stated that this behavior is manifested for the most part with citrus of early varieties or of early dates of harvest. The other line of evidence that points to the importance of redox level is that when "extra" ascorbic acid is added to citrus products before these are placed in frozen storage, the propensity toward development of COF has been observed to be lessened. This observation constitutes something of an enigma, however, because there is abundant evidence that the natural ascorbic acid of citrus remains something like 96% unoxidized in frozen storage *(11, 14)*. Why, then, is the addition of "extra" ascorbic acid a deterrent? It may be that the very considerable difference in redox level which exists between a sample which is 96% and one that is 98% in the reduced state could make a critical difference in the development of COF. It is only necessary to recall the S-shaped form of a redox curve and the steepness of the potential curve as it approaches the condition of pure reductant, to appreciate the force of this argument. Granting that the preponderance of the evidence points to redox level as being sometimes a controlling factor in this problem, the question remains as to the mechanism by which the redox level exerts its effect. The point is made by some observers, also, that the marked package-to-package variation sometimes encountered in material from the same batch, as described above, cannot plausibly be ascribed to any obvious differences in exposure to oxygen before freezing. Such observers are inclined to believe that factors other than oxidation are also important, and certainly it would seem that these sample-to-sample variations must be due to the accumulated effect, in a slow process during frozen storage, of subtle and perhaps analytically minor differences in the conditions which prevail.

Simulation of both tallowiness and malodor by use of components from grapefruit seed-oil. An hypothesis which has been found to be quite tenable is that both malodor and tallowiness are due to transitory intermediates in the biosynthesis of lipids. To check this hypothesis, which will be discussed more fully in the next section, it seemed possible that one might treat citrus lipid material in such ways as to "back-track," in a sense, upon this biosynthesis, and thus obtain materials which would be identical with, or similar to, the actual organoleptically-perceived intermediates. The lipid materials under consideration are those known to exist in very low concentration in the fruit pulp itself. In the case of orange this concentration has been found by Swift and Veldhuis *(21)* to amount to about 0.1%. However, since citrus seeds offer a much more convenient source of citrus lipid than does the pulp, and since it was hoped that for the present exploratory purpose no vital difference should exist between the two types of lipid, we have used grapefruit seed-oil throughout. We do not believe that the seeds, as such, have anything to do with COF.

Grapefruit seed-oil, freshly extracted from the dried ground seeds with Skelly B, ether or acetone, may be used. To avoid bitterness in subsequent taste tests, the oil is extracted thoroughly with 95% ethanol to remove the bitter component, limonin. In the first phase of the work, the oil was saponified by the use of glycerol and sodium hydroxide, diluted with water, acidified by sulfuric acid and distilled, all as specified in the official A.O.A.C. method for the determination of Reichert-Meissl and Polenske numbers. There remains behind,

in the distilling flask, a considerable amount of white water-insoluble material which may be termed the "non-volatile" acids, although much of this material will distil over with the steam if the distillation is prolonged.

Following is a summary of the taste-tests which were carried out in Florida during the spring season of 1955:

Tallowiness. The tallowy part of COF is usually best perceived as a lingering sensation after the juice is swallowed. Numerous individual taste-tests have shown that the tallowiness of COF may be quite well reproduced in normal juice by dispersing in such juice the "non-volatile" higher fatty acids obtained as described above. Dispersing the unsaponified seed-oil itself in juice does not produce this effect; this was shown previously by other workers and confirmed in the present investigation. The free fatty acids are necessary, but on the other hand it seems that the lingering quality of the tallowiness is best simulated if the fatty acids are dissolved in some of the unsaponified oil before dispersion in the juice. The added lipid material is dispersed in a portion of the juice by use of a Waring blender for at least 30 seconds, then diluted with the normal juice according to the perceptions of the observer. One cannot count on getting a "tallowy" response at a concentration much less than about 0.1% of the "non-volatile" acids, usually dissolved in about its own weight of seed-oil, but it is obvious that the degree of dispersion is very important. When the tallowiness factor is investigated by taste-tests with individual observers, so that the experimenter can adjust the "concocted" sample to simulate best the COF-tallowiness of the observer's experience, the results would seem to leave little doubt that the tallowiness of COF is in fact due to the presence of free higher fatty acid in abnormally high proportion; it does not seem possible that any completely different type of component could simulate this effect so well. However, the evidence of the present work along this line is confined to organoleptic evaluations of synthetic systems in the way just summarized. The conclusion has not been confirmed by analyses of the lipid components isolated from authentic COF samples to determine their free fatty acid content.

Malodor. That the tallowiness factor of COF can be plausibly attributed to higher free fatty acids is not surprising, but it was more unexpected to find that the aqueous phase of the Reichert-Meissl distillate, obtained as described above and stirred into normal citrus juice, was considered by competent observers to simulate, to a degree, the malodor of COF. Some observers used the term "castor-oil" without qualification to describe the resultant odor effect, but most observers were able to distinguish "true COF" from "synthetic COF" quite consistently. Even these observers, however, for the most part gave assent to a general similarity in odor quality between the two types of sample. Some of the flavor tests upon which these conclusions are based were carried out by the addition of the Reichert-Meissl distillate alone, but in other tests the tallowy "Non-volatile" acids were added as well. It seems safe to say that for the average observer the resemblance to actual COF was increased by using the Riechert-Meissl solution conjointly with the tallowy acids as addends. Even though the consensus was that the reproduction of the malodor was not exact, the resemblance cannot be ignored. Much further work, which will be described in a later section, has been carried out at Barrington during the past year to get further information as to the character of this malodor. It is suggested that the precursor of the malodor is a minor constituent of the seed-oil which is a residual intermediate in the biosynthesis of lipid, so that the malodor, as well as the tallowiness, is produced from the seed-oil by "back-tracking" upon its biosynthesis.

Biosynthesis of lipid as explanatory of COF. A second line of evidence, which suggests that COF is attributable to transitory stages in the biosynthesis of lipids in citrus pulp, derives from the correlation of the effect with physiological immaturity of the fruit. Figure 1, reproduced from Bonner [b] *(1)*, is a representation of the data

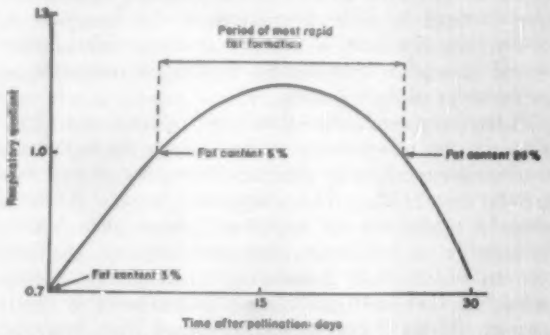

Figure 1. Respiratory quotient of developing castor fruits in relation to fat accumulation. (After Burr and Miller and reprinted, by permission, from J. Bonner, Plant Biochemistry, Academic Press, Inc., New York, N. Y.).

of Burr and Miller *(4)* and is illustrative of the generalization that most of the lipid of a fruit is produced during that immature stage of development in which the respiratory quotient is greater than unity. Other generalizations that are pertinent to the present problem are summarized by Bonner as follows: "fat is synthesized from an oxygen-rich precursor such as carbohydrate"; "it is clear that free fatty acids are first manufactured and that formation of glycerides follows as the seed matures"; "fat is formed in the organ itself, rather than transported into it from the leaf"; and "fat formation persists for a short time after removal of the organ from the plant."

In attributing the tallowiness of COF to new free fatty acid formed from sugar during the cold storage, rather than to fatty acid formed by lipolysis of preexisting fats during storage, one certainly has mass-action considerations in his favor in the case of citrus materials since the sugar content is so high and the fat content so low. If it is postulated that the enzymatic conversion of fatty acid to glyceride is more inhibited by the low temperature than is the enzymatic conversion of sugar to fatty acid, it would follow that the content

[b] For permission to reprint this figure, grateful acknowledgment is made to the authors and to the Academic Press, Inc., publishers.

of free fatty acid could build up in frozen storage to levels higher than those attainable in the normal process at ordinary temperatures. The organoleptic evidence as to the tallowiness factor, described above, would suggest that this may indeed be the case, but no quantitative data to establish this has been obtained in the present work.

However, Lee, Wagenknecht and Hening *(13)* have shown that very large increases in acid number of the extracted lipid are encountered in the early stages of the frozen storage of a number of raw vegetables. This increase in free acid content of the lipid must come either from lipolysis of pre-existing fats, or from the biosynthesis of new fatty acid as postulated herein. These authors imply that they have considered only the former hypothesis, but we are emboldened to suggest the biosynthesis hypothesis as applicable in their case as well, because their vegetable products would surely be considered to be physiologically immature and hence characterized by a tendency toward the biosynthesis, rather than the lysis, of fat. No evidence exists which would make the biosynthesis hypothesis untenable as explanatory of their results.

It has been established that intact cells are not necessary for the biosynthesis of lipid, since the enzymatic mechanism is intact in structural elements of less than cellular size *(15)*. The enzymatic process therefore may be postulated for expressed citrus juice just as reasonably as for intact grapefruit sections. A more serious objection to postulating an enzymatic process would be that COF is certainly encountered in "early orange add-back" concentrate, prepared from "enzyme-inactivated" juice, and to which no unheated citrus juice has been added subsequently. However, "enzyme-inactivation" refers only to an inhibition of the action of pectinesterase, and is a relatively mild pasteurization. A thorough investigation of the relationship of conditions of pasteurization to the incidence of COF in subsequent frozen storage has not been carried out, so far as the authors are aware. Then, too, there is a reluctance in the citrus industry to apply a full pasteurization.

In canning or freezing immature vegetables, a full blanch can be given and the malodorous products formed by enzyme action during blanching can be discarded in the blanch-water; this is impossible in the case of citrus products and the only alternative is to utilize, exclusively, fruit of physiological maturity in which the enzymes are relatively inactive. Vegetables are most succulent and flavorful when immature; citrus products, when mature. Probably the COF problem in citrus is not unique, but in some respects it is analogous to flavor problems encountered in the frozen storage of other fruit and vegetable products.

The observations indicating that redox level can be a limiting factor in the development of COF can be harmonized with the biosynthesis hypothesis by paraphrasing the discussion by Breusch *(2)* of carbohydrate-fat conversion processes. Undeniably, the conversion of a sugar (hexose) to a saturated fatty acid, say stearic acid, is a drastic reduction. Yet the biochemical evidence indicates that this reduction must be energized by the oxidation of some of the sugar to carbon dioxide. The mechanism of energy transfer need not enter into the discussion, for present purposes.

If sugar is completely oxidized to carbon dioxide and water by respiratory processes:

$$\text{I. } C_6H_{12}O_6 + 6\,O_2 = 6\,CO_2 + 6\,H_2O$$

the respiratory quotient (ratio of CO_2 produced to O_2 consumed) is unity, and no sugar whatever is converted to fat, or the "conversion coefficient" [the term was coined by Kleinzeller *(12)*], is zero. But it is perfectly possible to write and balance an equation which would represent a spontaneous conversion of hexose to stearic acid without the intervention of any extraneous oxidation:

$$\text{II. } 13\,C_6H_{12}O_6 = 3\,C_{18}H_{36}O_2 + 24\,CO_2 + 24\,H_2O.$$

In such a hypothetical case the respiratory quotient would be infinity (since no oxygen is consumed) and the conversion coefficient would be the percentage of the carbon that appears as fatty acid, or $54/(54 + 24) = 69.2\%$. However, the situation as expressed by Equation II is not experimentally realizable. The highest efficiency actually attainable in the conversion of sugar to fat would be that corresponding to the highest respiratory quotient encountered in a real situation. The respiratory quotient is all one needs to formulate and balance an equation for the process. The highest value cited by Breusch was that of 1.8 obtained by Kleinzeller. Accordingly, one may write:

$$\text{III. } 6\,C_6H_{12}O_6 + 10\,O_2 = C_{18}H_{36}O_2 + 18\,CO_2 + 18\,H_2O$$

which shows that the conversion coefficient corresponding to this respiratory quotient in the assumed simplified situation would be 50%. Kleinzeller's actual yield of fat was 26.3%. Breusch also cites the work of other investigators as evidence of the conclusion that a non-oxidative process such as that of Equation II does not take place. Although the ultimate oxidant in the overall enzymatic process would be oxygen itself, it is obvious that intermediate oxidants may be the ones of critical importance in the system under investigation, so that it may be just as significant that added ascorbic acid inhibits the development of COF as that the "Blendoring" with air appears to augment it.

Further investigation of the malodor producible from grapefruit seed-oil. The malodor contained in the Reichert-Meissl distillate as described above is doubtless complex and includes a contribution from the odor of the lower volatile and water-soluble acids. However, when the oil was saponified by barium hydroxide, so that the alkaline solution was distillable without foaming, the aqueous distillate was remarkably similar in its most characteristic aspects to the distillates obtained from acid systems. It was evident that the substances of most pronounced odor were neither acids nor bases.

It soon became evident also, that the precursor of the malador is a substance or substances present only as very minor constituents of the oil. Proportions of $Ba(OH)_2$, as low as one percent of that requisite for complete saponification, sufficed to produce from a given

sample of oil all of the malodor that it was capable of yielding. This led to an endeavor to isolate, or at least concentrate, the precursor by chromatography of the oil through alumina columns. Skelly B, trichloroethylene, ethyl ether, and methanol were used in that sequence as solvents. Each eluate was carefully evaporated nearly to dryness, the residue treated with $Ba(OH)_2$, heated under reflux, extracted with ether and the ether solution carefully evaporated on salt plates to be ready for examination for infra-red spectrophotometry. Usually very small amounts of waxy residues were observable to the eye. After much work it was recognized that the spectra observed were attributes of the waxy materials and that the odoriferous substances were present in such extremely low concentration as not to register significant effects in the spectra. However, it can be said, on the basis of olfactory perception alone, that Skelly B carried the greater part of the oil through the column leaving behind the precursors, which then appeared in the trichloroethylene and ether eluates. It was clear that the nose is far more sensitive than the infrared spectrophotometer.

It was then found that the best technic for developing a characteristically "castor-oil-like" odor from grapefruit seed-oil is by the use of dehydrating agents. Good results have been obtained by the use, at room temperature, of phosphorus pentoxide or of a cooled mixture of 85% phosphoric acid and phosphorus pentoxide. Concentrated sulfuric acid will do the same thing, but must be used in exceedingly small proportion. If more than a minute amount is added, some of it is reduced to sulfur dioxide which obscures all other odor. It is well known that phosphoric acid and phosphorus pentoxide are not oxidizing agents. In fact, even reagent grades of these substances contain traces of the corresponding derivative of trivalent phosphorus so that a tendency toward reduction, rather than oxidation, is to be expected. Dehydration to produce the same odor can even be effected by the use of calcium oxide (small hard granules) or of anhydrous magnesium sulfate, though with these agents the odor is best observed after slight warming. It seems to be a safe conclusion that the chemical reaction by which the odor is produced from the precursor is a dehydration. The standard procedure used to produce the odor in the work described hereafter is as follows. Dried grapefruit seeds are dropped into a dry Waring blender which chops them up expeditiously. The seed-coats are removed by screening and the remaining coarse powder is extracted, in a Soxhlet, by acetone. Most of the acetone is distilled off and part of the rest is evaporated at room temperature. Some of the turbidity then present is water-soluble and is removable by a single water extraction. Two-hundred grams of the oil is then extracted with four successive 25-ml. portions of 95% ethanol. This removes the bitter component limonin; the additional use of dilute potassium hydroxyide *(16)* is not necessary. The oil is then filtered and is a clear yellow product having only a slight pleasant nutty odor. It is decolorized and rendered practically odorless by filtering the two-hundred grams, with suction, through a six-inch column of a mixture of one-third Mallinckrodt's activated carbon and two-thirds Filter-Cel. Though this treatment may possibly remove some of the precursor, it affords the greatest possible odor contrast producible by the dehydration reaction. The principal objective was to attain unambiguous qualitative results, rather than to estimate the maximum possible yield.

Ten to 20 g. of the odorless oil is added to a mixture of 100 ml. 85% H_3PO_4 and about 20 g. of P_2O_5 in a Corning No. 31770 gas-washing bottle equipped with a fritted glass dispersion tube. Nitrogen is passed through the mixture and thence through a series of three Peligot absorption tubes, each containing about 5 ml. of solvent. The solvents used were absolute ethanol, isopropanol, or water.

The alcoholic solutions so obtained give positive Schiff tests for aldehyde and also give the Hiltner test *(17)* as modified by Wearn and coworkers *(22)*. The latter test is considered to be specific for alpha, beta unsaturated aldehyde or 2-ene-1-al. It is confirmatory of the presence of this kind of aldehyde that positive tests have also been obtained with the pentacyanommineferroate reagent, as described by Feigl *(7)*. However, the latter test, involving the formation of thioaldehyde, is not so sensitive as the other tests and has not been positive in every instance. When water is used as the absorbing solvent, the solutions reduce ammoniacal silver. The aqueous solutions do not give a positive thiobarbituric acid test except in the presence of a trace of cupric ion, but when this ion is added as part of the analytical procedure a positive test, though weak, is invariably obtained. This behavior, with regard to the thiobarbituric acid test, has been shown by Patton and Kurtz *(18)* to be characteristic of alpha, beta unsaturated aldehydes. It would appear to be a safe conclusion that the "castor-oil" or "cardboard" odor developed by dehydration of an odorless grapefruit-seed-oil, contains alpha, beta-unsaturated aldehydes. Such aldehydes certainly would not be odorless; therefore they must be components of the odor blend which is described by such adjectives. It is probably significant that the word "cardboard" is also used to describe a flavor effect in milk which has been shown to be correlated with the presence of such aldehydes. This will be discussed further below. However, we should not conclude that such aldehydes are alone responsible for such odor effects, nor that all 2-ene-1-als have an odor of this character.

Spectrophotometry of the Hiltner-Wearn and thiobarbituric acid tests. A General Electric double-beam recording spectrophotometer was used to make more definite the results of both tests. Two-centimeter cells were used in all cases. It is almost impossible to get a completely colorless blank in the Hiltner-Wearn test because of difficultly removable impurities in the m-phenylenediamine reagent; therefore it is a great advantage to have the use of a double-beam instrument and to put the blank in the comparison light-path. Different 2-ene-1-als give colored derivatives in this test which differ as to wave-length of maximum absorption. Therefore the test will differentiate between such unsaturated aldehydes if the test is made on each separately, but it has been found that if the m-phenylenedia-

mine reacts with a mixture of two aldehydes the reaction product appears to involve both and the absorption band will lie between those which are characteristic of the derivatives of the two components. In Figure 2,

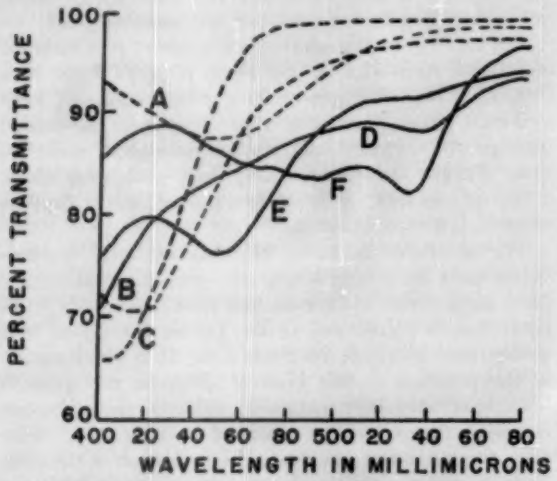

Figure 2. Spectrophotometry of two color tests for unsaturated aldehydes.
Hiltner-Wearn Test (Dotted Curves)
A. Transferred by N_2 from phosphoric-treated seed-oil.
B. Transferred by N_2 from grapefruit essential oil.
C. Crotonal at 1.5 micrograms per milliliter.
Modified Thiobarbituric Acid Test (Solid Curves)
(Positive only when Cu^{++} is present during the test).
D. Transferred by N_2 from phosphoric-treated seed-oil.
E. Transferred by air from phosphoric-treated oxidized seed-oil.
F. Crotonal at 2.2 micrograms per milliliter.

curve A shows the absorption centering about 450 mμ, which has been found in several different experiments to characterize the Hiltner-Wearn derivative of the nitrogen-wafted substance produced by dehydration of grapefruit-seed-oil. Curve C shows the characteristic absorption of the derivative of crotonaldehyde, centering about 405 mμ. It is obvious that the 2-ene-1-al of the COF malodor is not crotonal. Nevertheless the the absorption of 1.5 μg. of crotonal in Curve C may be used as a rough quantitative measure of the extremely low concentration of 2-ene-1-al represented by Curve A; yet the odor effect is quite easily perceived. Curve B was obtained by passing nitrogen through a dilute suspension of grapefruit essential (peel) oil in water, and thence into the absorption tubes containing alcohol. This curve shows that the essential oil also contains 2-ene-1-als, but these are assuredly not the same as the COF malodor. It is our hope that the Hiltner-Wearn test, absorbing at 450 mμ, can be recommended as useful in survey work comparing various citrus samples as to the presence of COF, but Curve B (as well as a limited amount of other experimentation) shows that troublesome interference may be expected with commercial or experimental packs which contain much essential oil.

Turning to the thiobarbituric acid (TBA) test, Curve D of Figure 2 shows the characteristic absorption of the TBA color given by the aqueous solution of substances wafted out of phosphoric-dehydrated grapefruit seed-oil by nitrogen. Curve F shows the test obtained with an aqueous solution of 2.2 μg. of crotonal. In the present work the test was applied by mixing equal volumes of the aqueous sample, 0.1 molar citrate buffer pH 3.5, and 0.01 percent aqueous thiobarbituric acid. To 10 ml. of this mixture is added 0.1 ml. of a one percent solution of $CuSO_4 \cdot 5H_2O$, and the reaction mixture is then immersed in boiling water for 15 minutes. Control mixtures of the same components, except that copper is absent, remain strictly colorless, and this is also true with the crotonal standards. It will be observed that the TBA derivative of both crotonal and the grape-fruit seed-oil product absorb at about 535 mμ. This is a general characteristic of the TBA test as cited in the literature describing a wide variety of applications of this test. The non-variance of this absorption band in the TBA test is in marked contrast with the variation in absorption given by different 2-ene-1-als in the Hiltner-Wearn test. Curve E is of especial interest. This shows what is obtained when the phosphoric mixture with grapefruit seed-oil is allowed to stand overnight in the presence of oxygen and a trace of cupric ion, and the volatile products are then wafted out into the test solution with a current of air instead of nitrogen. Further cupric ion is added in the testing procedure, as described above. This curve E bears a close resemblance to the curve obtained with 2-heptenal by Patton and Kurtz. A slight absorption band at 535 mμ remains, but an additional absorption band centering about 450 mμ is now present. It seems plausible to assume that under such conditions actual oxidative disruption of major components of the oil, such as unsaturated fatty acids, has taken place. This also produces 2-ene-1-als, but these are different from the unsaturated aldehydes which are considered to constitute the primary COF malodor, and are more representative of the aldehydic components which constitute a part of ordinary oxidative rancidity.

Biosynthesis of lipid as explanatory of COF malodor. Figure 3 sets forth diagrammatically the biosynthesis of lipids in plants, from sugar via acetaldehyde and aldol-like precursors. Much recent work on lipid

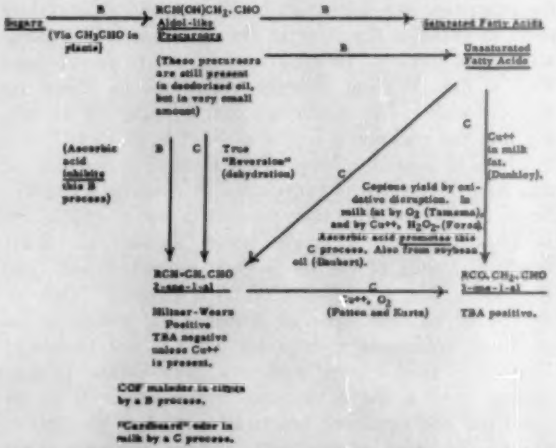
Sugars
B
(Via CH_3CHO in plants)
$RCH(OH)CH_2.CHO$
Aldol-like Precursors
Saturated Fatty Acids
(These precursors are still present in deodorized oil, but in very small amount)
Unsaturated Fatty Acids
(Ascorbic acid inhibits this B process)
True "Reversion" (dehydration)
Copious yield by oxidative disruption. In milk fat by O_2 (Tamsma), and by Cu^{++}, H_2O_2 (Forss). Ascorbic acid promotes this C process. Also from soybean oil (Ihnbert).
Cu^{++} in milk fat. (Dunkley).
$RCH=CH.CHO$
2-ene-1-al
$RCO.CH_2.CHO$
1-one-1-al
Cu^{++}, O_2
(Patton and Kurtz)
TBA positive.
Hiltner-Wearn Positive
TBA negative unless Cu^{++} is present.
COF malodor in citrus by a B process.
"Cardboard" odor in milk by a C process.

Figure 3. Schematic representation of certain biochemical (B) and chemical (C) changes affecting lipids.

biosynthesis in animals has led to other seemingly different mechanisms but, as Hilditch has pointed out *(10)*, the aldol mechanism remains the best in regard to plant metabolism. Plants can synthesize unsaturated as well as saturated acids (Hilditch suggests that the two mechanisms may differ) and plants can do this in every structural part of the plant. It has been shown by Kleinzeller *(12)* and others that acetaldehyde in plant tissue appears to be a precursor of lipid, and it is interesting that Burger and co-workers *(3)* found that the addition of acetaldehyde (though not acrolein) to the brew produced a "cardboard" off-flavor in beer. The oxidative flavor occurring in milk is often called "cardboard," as by Forss and co-workers *(8)* who attributed this odor effect to the whole series of 2-ene-1-als ranging from 4 to 11 carbon atoms, based on chromatographic identification of the 2,4-dinitrophenylhydrazones. Tamsma *(20)* used ultra-violet spectrometry to come to a similar conclusion, although he felt that other compounds, perhaps non-conjugated enals, might be even more important. The conditions established with milk-fat by these workers to obtain this effect were certainly oxidative. Thus, although Forss worked at 2° C., he always used cupric ion and exposure to air, sometimes in the presence of ascorbic acid which acted as an oxygen carrier and promoted the effect. In some cases hydrogen peroxide was also added. Certainly these changes are chemical, distinguished in Figure 3 by the letter C, to distinguish them from biochemical or physiological effects distinguished by the letter B. Let us suppose that a "cardboard" like organoleptic effect is a property of certain enals, no matter how they may be formed, and then let us contrast the milk-fat situation with the COF situation. Let us consider for example an unruptured grapefruit section, developing this odor at —18° C. (0° F.). Here ascorbic acid inhibits the effect. Here we believe that the *primary* COF odor is due in part to the enals which arise inside this grapefruit section by a biochemical or B dehydration of the aldol-like precursors involved in lipid biosynthesis. We suppose that freshly extracted grapefruit seed-oil contains very small amounts of these aldolic precursors, which are themselves odorless and are partially retained in the oil after deodorization. When the deodorized oil is now subjected to a non-oxidative chemical or C dehydration, enough enal is formed to suggest a "reversion" to an odor somewhat akin to the natural odor of the freshly-extracted non-deodorized vegetable oil. The literature pertaining to the odors of vegetable oils seems to leave the borderline between "reversion" and "incipient oxidative rancidity" somewhat indistinct, and of course actual samples of an oil must often exhibit the results of both effects mixed in almost inextricable confusion. The work of Daubert and co-workers *(9)* on the "heat-reversion" of soybean oil can be cited as showing that sometimes an odor can be produced by a non-oxidative process that is at least as bad as one produced by an oxidative process. As Figure 3 shows, the thought is that even though enals may be present in both types of malodor, the precursors are different. Only oxidation can disrupt a long-chain unsaturated fatty acid to produce a malodor, but the *primary* COF malodor, at least, is related to the natural odorants present in all vegetable oils as a result of the same biological processes by which the oils themselves are produced. These natural odors are often undesirable, as in the case of castor oil itself, and even in the common edible vegetable oils the natural odorants are undesirable if present in excess.

It is remarkable that the Hiltner-Wearn test has not been used in investigations of the oxidized milk problem, though Dunkley *(5)* as well as Patton and Kurtz *(18)* have used the thiobarbituric acid test. The Hiltner-Wearn reagent has been used qualitatively in connection with the reversion of soybean oil.

An incidental observation. It is odd that an organoleptic effect capable of being described as "castor-oil-like" would also at times be described as "cucumber-like," but this was the case, both with the nitrogen-wafted effluent from phosphoric-treated grapefruit seed-oil and with the barium hydroxide distillates mentioned above. It may be worth mentioning, therefore, that Takei and Ono *(19)* have investigated the fragrance of cucumber and have ascribed it to a mixture of 2,6-nonadienal and the corresponding diene alcohol. Similar dienals have been found among the odorous components of other fresh vegetation, and these facts are cited here as showing that the biosynthesis of such enals is certainly possible, even though it may not always be associated with the biosynthesis of lipid.

Use of the Hiltner-Wearn and TBA tests in the evaluation of COF. In preliminary work in Florida in the spring of 1955, a double-puncturing device was used to pass nitrogen through the headspace of six-ounce cans of frozen concentrate, thence directly through a Peligot tube containing the Hiltner-Wearn reagent. Weak positive tests were obtained which seemed to correlate with organoleptically-observed COF. But no spectrophotometer was available and the color was so weak that only a few observations were made and little faith was placed in the result until it was found, months later, that the castor-oil-like effluent from phosphoric-treated seed-oil gave definitely positive tests. In recent work in which the double-beam spectrophotometer was available, the headspace gasses from certain samples of citrus concentrates have given Hiltner-Wearn derivatives showing weak absorption at 450 mμ. Furthermore, weak thiobarbituric acid tests have also been obtained (provided cupric ion is used in applying the test) with headspace gasses from citrus concentrates. However, the organoleptic symptoms of COF in these recent samples were weaker and more questionable than in the samples that were available a year ago. Although it is possible that these tests could now be useful in the evaluation of citrus packs, and well worth trying in view of the uncertainties of organoleptic evaluation in this problem, it would certainly require much more extensive survey applications in the field before one could arrive at valid judgment in the matter. The interference by citrus essential oils has already been mentioned.

Practical need for a field index of true maturity. The work herein cited has been carried out in an effort

to arrive at an understanding of the mechanisms of the COF effect. If the conclusion reached is right, that the underlying cause of the primary effect is a lipid biosynthesis characterizing physiologically immature fruit, then it would seem that a simple and reliable field evaluation of the physiological maturity factor so as to avoid COF, would be of even greater value to citrus processors than a color test to measure the intensity of the effect after it appears. A measurement of physiological maturity would tell the processor when he could begin to harvest for frozen pack, and also how to select fruit for freezing while utilizing the less mature fruit in other ways. Perhaps the procedure of Eddy *(6)* in measuring the absorption rate of oxygen in citrus juice could be modified to measure the respiratory quotient well enough for practical purposes.

SUMMARY

The conditions are summarized under which an off-flavor, termed the "COF effect" and characterized by a "tallowy" mouth-feel and a "cardboard" or "castor-oil-like" malodor, may appear during the frozen storage of citrus products. Evidence leads to the conclusion that physiological immaturity is fundamentally causative of the phenomenon. Talloviness can be well reproduced by the device of dispersing in normal citrus juice the higher fatty acids obtained by saponification of debittered grapefruit seed-oil. Malodor can be simulated by substances, including alpha, beta unsaturated aldehydes, obtained from minor components in grapefruit seed-oil by chemical treatment that constitutes dehydration, rather than oxidation. Evidence is presented in support of the view that both tallowiness and malodor are of biochemical origin and represent stages in the biosynthesis of lipid from sugar. This biosynthesis is known to be most active in immature fruit. Although the COF effect is modulated by redox level, reasons are given for differentiating it from a simple chemical disruption of fats by oxidation. Malodor is believed not to be ascribable to actual oxidative disruption of fats, but to be correlated with the odorous components, disagreeable at times, which characterize unrefined vegetable oils in general and are products of the same biosynthetic processes that produce the fats themselves, in plants. The alpha, beta-unsaturated aldehydes constituting at least part of the malodor are present, not because some of the fats have been oxidized, but because the biochemical conversion of sugar to fatty acid, which is actually a drastic reduction, has failed to go to completion.

A basis exists for the concept that this lipid biosynthesis is significant not only for frozen citrus, but for frozen vegetable products as well if these are insufficiently blanched. Since in the case of citrus it is impossible to blanch and subsequently to eliminate the products of the enzymatic action which takes place during blanching, the best recourse would be to await the attainment of that stage of physiological maturity in which the enzymes are relatively dormant. It is pointed out that a measuremement of respiratory quotient might afford a useful criterion of physiological maturity.

Acknowledgments

Mr. Arthur F. Diesing carried out the experiments on the chromatographic separation of grapefruit seed oil into various fractions, and Mr. Paul W. Hardy operated the General Electric recording spectrometer in connection with the Hiltner-Wearn and thiobarbituric acid tests. This help is gratefully acknowledged.

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Changes In the Water-Holding Capacity And Cationic Shifts During the Heating and Freezing and Thawing of Meat as Revealed By a Simple Centrifugal Method For Measuring Shrinkage[a, b]

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SHRINKAGE AND WATER-HOLDING CAPACITY of meat and particularly of processed meats have always been of great concern to meat packers and hotel and restaurant operators. Primarily this concern was from the standpoint of yield of product. More recently studies on consumer quality attributes have revealed that such important factors of tenderness, texture, drip on freezing, etc., are controlled by the water-holding capacity of meat proteins *(1, 10, 11, 12)*. The importance of certain ions and the water-holding capacity of proteins in functioning muscle is well-known to biochemists and physiologists. Studies along these lines but directed toward problems of meats have been intensively studied by Grau and Hamm *(2, 3, 4, 5, 6, 7)* at the Bundesforschungsanstalt für Fleischwirtschaft in Kulmbach, Germany, as well as by the authors and their associates.

An attempt has been made to shed some light on the effect of temperature of cooking on the water-holding capacity and on the ion-protein interactions in meat. This type of study follows directly from work previously reported concerning post mortem shifts in ion-protein relationships, changes in water holding capacity and tenderization *(10, 11, 12)*. The technique for measuring shrinkage *(12)* has been simplified so that no transfers of meat or juice are necessary. Furthermore, sufficient meat and juice are obtained for study of ion shifts taking place during the heating and protein denaturation of cooking. Thus, it has been possible to obtain basic information of importance to meat processing problems involving heating. The techniques have also been applied to the study of drip losses on freezing as affected by post mortem aging of meat and the addition of salt.

METHODS

Except where noted, the procedures of sampling and analyses are those previously reported from this laboratory *(8, 9)*.

Sodium, potassium, calcium, and magnesium were determined as described by Arnold *et al. (1)*.

Determination of the water-holding capacity of fresh and cooked meat. The method for the determination of moisture loss on cooking meats used previously *(12)* has been developed further into a rapid and accurate method for measuring the juices lost on cooking, freezing and thawing and the like. The procedure finally adopted requires a specially constructed centrifuge tube, constant temperature water bath, a centrifuge, and a magnifying glass.

The heavy Pyrex tubes were especially made by the Corning Glass Works. Figure 1 shows the empty tube on the left, the tube with sample ready for heating, and the tube at the end of the determination with residual meat and juice. The tubes are 180 mm. long overall. The top section is 30 mm. in diameter and the bottom is 20 mm. The smaller section is graduated to 12 ml. at 0.1 ml. divisions and is approximately 75 mm. in length. A loose fitting, very coarse fritted glass disc rests in the tube at the junction of the small and large sections. Tests are carried out as follows, using as many tubes as may be centrifuged simultaneously.

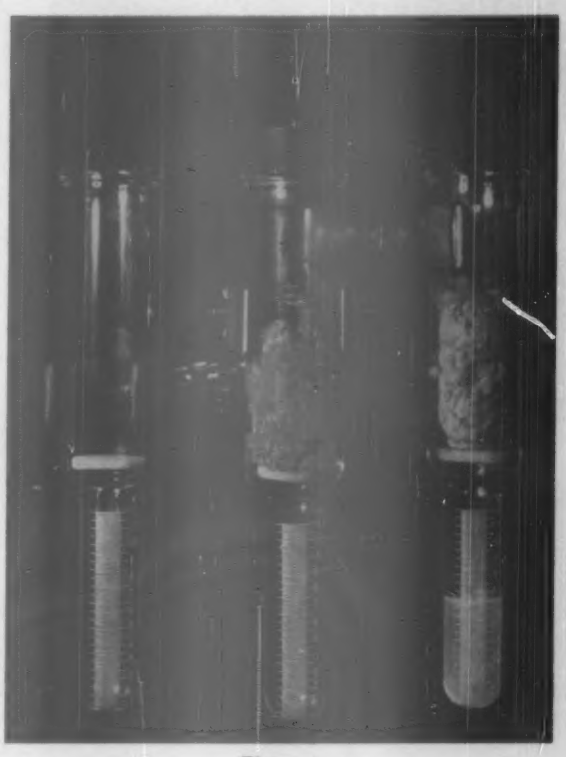

Figure 1

Approximately 25 g. of thoroughly ground and mixed or unground meat is weighed and placed in the upper portion of the tube with the sample resting on the glass disc. The tube is closed with a rubber stopper holding a glass capillary which acts as a vent. A number of such tubes may then be immersed into a water bath to depth of about 9/10 of the tube length for 30 minutes. For routine shrinkage tests 70° C. is recommended, whereas 40° C. works best for uncooked meat where such things

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as drip losses are to be measured. When it is desired to run tests at the temperature of boiling water, the tubes should be removed from the bath for about one minute after the first 15 minutes. This procedure permits some of the juice to drop into the lower portion of the tube and to expel some of the air thus preventing meat samples from being lifted against the stopper and losing juice via the capillary. The heating is then continued until the 30 minutes has elapsed.

After heating, the tubes are immersed for 10 minutes in 20-25° C. water to cool. Solidification of fat should be avoided since it may interfere with centrifugation and with reading the volume of juice not fat at the completion of the test. They are centrifuged for 10 minutes at 170 x gravity and, using Model PR-1 International Refrigerated Centrifuge, at 1000 r.p.m. In case meat samples are heated only to 30-50° C., centrifugation is interrupted after 5 minutes and the sample is punched with a small glass rod to break up juice pockets which might develop. After centrifugation is completed the volume of juice is read using a magnifying glass. Liquil fat which in some instances may be expressed is not considered as part of the juice. This fat collects at the top of the graduated column and the aqueous layer may be read directly.

Percent of moisture loss is computed using the relation:

$$\text{Percent moisture lost} = \frac{\text{Ml. juice} \times F}{\text{Grams of moisture in sample}} \times 100$$

Moisture in sample may be determined by taking two 12.5 g. samples of the same meat and heating at 105° C. for 6 hours. If these duplicates agree, indicating satisfactory sampling, then the sum of the moisture loss is equal to the denominator in the above relation. F represents the fraction of the juice which is water. For juice expressed at 40° C. F has been found to be equal to 0.913 ± 0.002; at 70° C. F equals 0.951 ± 0.004.

After each use the tubes are washed with soapy water, rinsed with distilled water and acetone and wiped dry with a towel. The discs are blown with a stream of compressed air for 14-20 seconds rather than completely dried. If the filters are dried completely, they will retain approximately 0.1 ml. of juice during the following determination. When experiments are finished the discs are placed in dilute sulfuric acid to dissolve substances of meat juice adsorbed to the filters. The acid must be washed out before reuse.

Precision of the method. When heating is carried out at 70° C., duplicates usually agree to 0.1 ml. of juice 25 g. of meat. When lower temperatures are used or when samples are less homogeneous, more than duplicate determinations are required to get this accuracy.

It is appreciated that the method is rather empirical; yet it is reproducible and quite useful. The primary error appears to be the centrifugal force applied rather than time of centrifugation. Table 1 brings this point out and also indicates the reproducibility of duplicate determinations. Although not shown here, heating curves show that 30 minutes in the water bath is adequate. Readings should not be taken if fat is solidified prior to centrifugation. Solid fat blocks the flow of juice and makes reading of the aqueous layer difficulty. Although a 25-g. sample is recommended, sample sizes from 15 to 40 g. do not greatly alter reproducibility. It should be noted that ground meat releases somewhat less juice than unground meat from the same muscle. A number of trials on the same muscle indicates that approximately ⅙ (16%) less juice is expressed by the ground sample as contrasted to the unground sample.

TABLE 2

Effect of aging and the kind of muscle on the meat shrinkage —percent of total moisture expressed in juice

Animal	Muscle	pH		70° C.		80° C.		99° C.	
				Days Post-Mortem					
		2	15	2	15	2	15	2	15
Steer 3	Loin	5.40	5.50	.....	.....	45.7	41.0	52.1	48.3
Steer 4	Loin	5.40	5.48	.....	.....	43.8	41.6	49.9	43.5
Steer 5[1]	Loin	5.37	5.45	37.1	27.6	.....	.....	50.9	46.9
Steer 6[1]	Loin	5.30	5.40	36.7	33.2	.....	.....	50.5	47.0
Cow 4	Loin	5.37	5.61	.....	.....	43.8	35.7	50.4	48.2
Cow 4	Round	5.30	5.48	.....	.....	45.6	44.6	52.2	50.2
Cow 5	Loin	5.43	5.51	32.5	28.4	45.4	41.0	51.5	44.1
Cow 5	Round	5.39	5.43	35.2	31.1	44.0	39.4	51.2	46.2
Cow 5	Chuck	5.57	5.61	42.1	40.4	49.7	44.8	56.5	50.6

[1] Determinations made at 2 and 13 days post mortem, respectively.

The juice expressed by different muscles will vary and also the amount of juice lost on heating will vary with post mortem age. This is illustrated in Table 2, which in part confirms earlier reports that aged meat shrinks less than unaged meat *(12)*.

RESULTS AND DISCUSSION

Effect of temperature of heating on the water-holding capacity, on the pH of the cooked meat, and on the ionic shifts taking place during cooking was studied using meat with nothing added, meat with 10% 2.2 M. sodium chloride added (equivalent to 1.3% sodium chloride based on meat) and meat with an equivalent amount of distilled water added.

The loin muscle of a 5-year-old-cow, 8 days post mortem, was thoroughly ground and mixed. To one-third of the meat 10% of its weight of 2.2 M. sodium chloride was added and thoroughly mixed; to another third an equivalent amount of distilled water was added with thorough mixing; and the remaining meat was used with nothing added. Samples representing 25 g. of meat (27.5, 27.5, 25 g., respectively) were used for shrinkage or juice determinations as described above over the temperature range from 30°-100° C. (Figure 2). In general the amount of moisture lost increases with increasing temperature but not uniformly so. It is highly significant that in the temperature range of 55°-65° C. minima occur in these curves indicating that reactions are taking place to counteract loss of water by

TABLE 1

Effect of time and speed of centrifugation on the final amount of juice

Speed r.p.m.	RCF	Milliliters of juice released by 25 grams of beef											
		Temp. of water bath	40° C.[1]				70° C.[2]				99° C.[2] (boiling water)			
		Min.	0	10	20	30	0	10	20	30	0	10	20	30
1000	170xg		0.0	1.9	2.0	2.1	1.8	4.1	4.3	4.5	7.5	8.1	8.1	8.2
				1.8	2.0	2.2	2.4	4.3	4.4	4.6	7.4	8.1	8.1	8.1
1500	383xg		0.0	2.4	2.8	3.1	2.0	5.2	5.5	5.7	7.5	8.2	8.2	8.3
				2.5	2.8	3.2	2.5	5.3	5.6	5.8	7.4	8.2	8.2	8.2
2000	681xg		0.0	2.9	3.1	3.3	2.0	6.0	6.3	6.5	7.5	8.5	8.8	8.9
				3.0	3.2	3.5	2.2	6.2	6.2	6.5	7.5	8.6	8.9	8.9

[1] Cow, round muscle, 15 days postmortem, moisture 72.9%.
[2] Steer, loin muscle, 9 days postmortem, moisture 72.9%.

the proteins. Note also the hydration effect of added sodium chloride and the minimum is even more pronounced than with meat with added water or meat alone. It is of particular interest that these irregularities occur in the temperature ranges of interest to meat processors in certain smokehouse and cooking operations. Furthermore, these particular temperatures are the critical ranges in broiling and roasting certain meats—particularly beef. These curves help understand the nature of the toughening observed in well done meat as contrasted to medium or rare meat. The tenderizing effect of salt as improving the water-holding capacity of meat is well brought out in Figure 2.

The pH changes occurring during the heating of the meat are shown in Figure 3. The pH values of the residual meat and of the juice are shown for the control meat and for the meat containing added sodium chloride over the same temperature range. In decided contrast to the amount of juice expressed, pH of both meat and

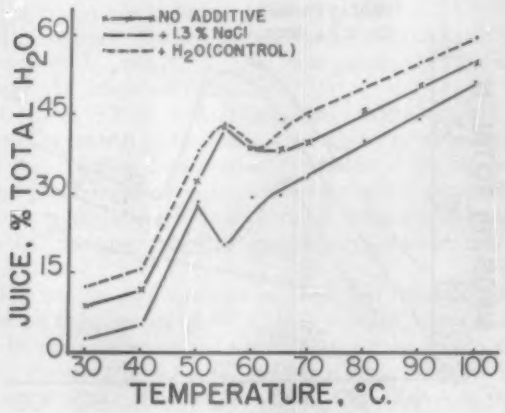

Figure 2

juice do not go through minima but show relatively uniform increases with temperatures up to 75°-80° C. The rapid increase in pH of both meat and juice between 40°-70° C. shows that reactions other than shifts in pH account for meat shrinkage curves shown in Figure 2. It should be pointed out that, whereas sodium chloride increases the pH of the meat at all temperatures, this is not true of the juices expressed. This indicates that sodium ions are preferentially adsorbed on the meat proteins as they are heated, while chloride ions are released into the juice.

Figures 4-7 show the corresponding cationic shifts produced by heating meat over the 40°-100° C. temperature range. Figures 4 and 5 give the amounts of sodium, potassium, calcium, and magnesium expressed as milliequivalents per 100 ml. of juice. If these data are recalculated as milliequivalents of cations released per one gram of total nitrogen in the meat, Figures 6 and 7 result. It can be seen that in the temperature range of 50°-70° C. potassium ions are absorbed by the meat, whereas magnesium and calcium ions are released. The addition of sodium chloride produced a very interesting effect—it increased the absorption of both potassium ions and magnesium ions with almost no effect on calcium ions. This experiment was repeated, again using a muscle from the round of another cow 15 days post mortem, and the results were identical. Much remains to be done to understand the full signifi-

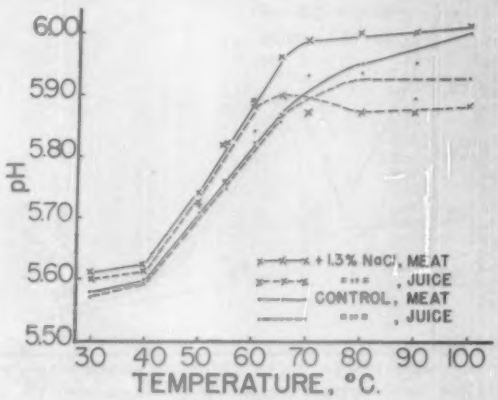

Figure 3

cance of the ionic shifts. Additional work along this line will be reported in a later paper.

In general, the foregoing results indicate that heat denaturation of muscle proteins begins at about 40° C. and is essentially finished at 70° C. Thus, 70° C. is sufficient for the determination of water-holding capacity of heat-denatured meat proteins, and 30°-40° C. can be used to study moisture losses in fresh meats or in frozen and thawed meats. (See below.) In the 40°-70° C. range, dynamic shifts involving potassium, calcium, and magnesium take place in such a manner as to promote hydration of meat proteins, and thus tend to counteract the dehydration usually associated with heat denaturation. This tendency is promoted by added sodium chloride. The foregoing results on pH changes with heating indicate heat denaturation of meat proteins may destroy some free carboxyl groups and perhaps some free sulfhydryl groups on the meat proteins.

The techniques described above for the study of heat denaturation have been applied to a preliminary study

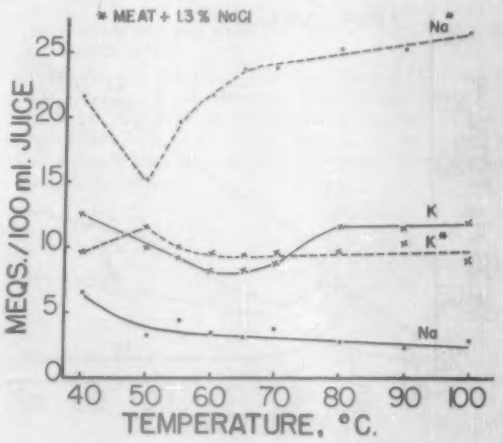

Figure 4

of drip losses after freezing and thawing in relation to post mortem age. Samples of meat were ground and mixed; part was used fresh and the remainder was put in glass containers and placed in a freezer at —20° C.

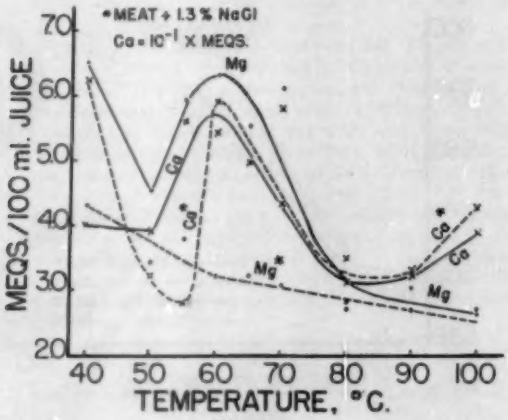

Figure 5

for one week. The frozen samples were then allowed to thaw in a 2° C. cold room overnight. After thawing and mixing, 25-g. samples were warmed at 40° C. to determine loss of juices. Table 3 shows the drip losses and pH values obtained from 7 different muscles representing 4 different animals at 2 and 15 days post mortem. The great advantage aged meat has in decreasing drip is obvious. Whereas at 2 days post mortem, drip losses ranged from 22 to 33% (ave. 27%), at 15 days drip losses were only three to 17% (ave. 11%). To be sure, this is an empirical test and the absolute values may not carry over directly to commercial practice, yet there is little doubt concerning the observation that drip losses are higher in fresh than in aged meat. Whether or not the difference is significant between moisture loss of aged unfrozen as compared with aged frozen meat, remains for further study. It is interesting that this difference is the same order of magnitude as that observed between fresh and aged unfrozen meat. There has been some commercial practice of freezing meat to decrease shrink in later processing.

TABLE 3

Effect of aging on the moisture lost on heating meat at 40° C. Before and after freezing and thawing

Animal	Muscle	pH of fresh meat		Juice, % of total moisture			
				Before freezing		After freezing	
		Days post-mortem					
		2	15	2	15	2	15
Steer 3	Loin	5.40	5.50	27.2	12.0	33.1	9.4
Steer 4	Loin	5.40	5.48	20.6	11.6	24.1	12.4
Cow 4	Loin	5.37	5.61	13.9	8.3	24.2	2.7
Cow 4	Round	5.30	5.48	19.4	17.2	23.8	16.7
Cow 5	Loin	5.43	5.51	22.7	12.0	33.2	5.5
Cow 5	Round	5.39	5.43	22.8	20.8	29.9	12.8
Cow 5	Chuck	5.57	5.61	8.5	14.6	22.4	16.5

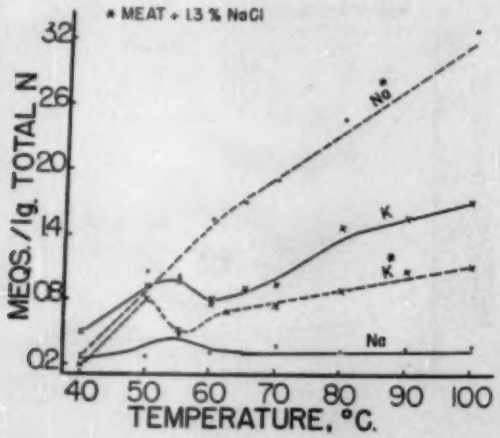

Figure 6

An earlier report from this laboratory indicated that added sodium chloride could decrease drip losses in frozen meat *(10)*. By using the foregoing technique for measuring drip losses this effect of sodium chloride is

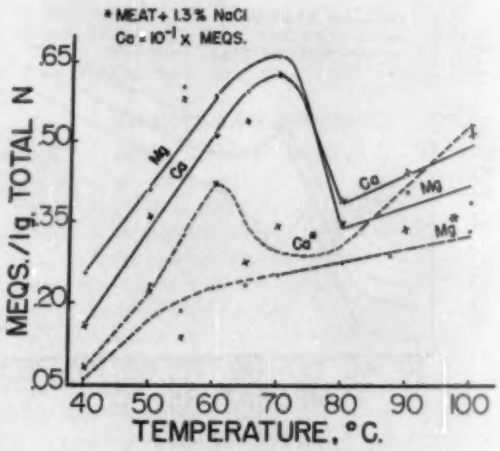

Figure 7

given in greater detail in Table 4. It is of interest that the minimum drip losses occur in the region of 5-6 M. in terms of the added solution or 3-3.5% based on the meat. This minimum has been observed in connection with meat heated to 70° C. and will be reported in greater detail in a later paper. It should be noted that, whereas the addition of sodium chloride increases the pH of meat before freezing, the meat tended to approach its original value after freezing and thawing. The pH of the expressed juice was increased also by the sodium chloride. This increase in pH was even greater than observed for the meat and furthermore was carried over into the drip from the frozen and thawed meat. It is of interest to note that the pH of the juice also tended to be lowered by the freezing and thawing.

SUMMARY

A simplified centrifugal method has been developed for the study of water-holding capacity (shrinkage) of meat. The technique has been applied to the study of the relationships of temperature of heating, added water, added salt, post mortem age and freezing and thawing on the water-holding capacity of meat. The method

TABLE 4

Effect of sodium chloride on the moisture loss and pH of ground beef heated at 40° C. Before freezing and after freezing and thawing

No.	Additive (Addit./meat = 1/10)	Juice, % of total moisture				pH (c)			
		Cow 6		Cow 7		Meat		Juice	
		a	b	a	b	a	b	a	b
1	No additive	12.8	28.0	12.7	25.7	5.56	5.55	5.61	5.57
2	H_2O (Control)	21.2	35.9	19.7	26.6	5.56	5.54	5.59	5.58
3	1 M NaCl	20.6	30.2	16.2	21.2	5.62	5.55	5.67	5.58
4	2 M NaCl	15.3	25.6	9.4	10.3	5.64	5.57	5.74	5.60
5	3 M NaCl	12.8	20.6	7.8	10.3	5.68	5.58	5.81	5.63
6	4 M NaCl	5.9	14.8	4.2	6.9	5.70	5.55	5.85	5.60
7	4.5 M NaCl	5.7	13.8	2.2	6.9	5.70	5.54	5.88	5.65
8	5 M NaCl	5.4	11.3	4.9	6.1	5.67	5.57	5.84	5.70
9	5.5 M NaCl	6.9	10.8	6.4	4.2	5.74	5.56	5.87	5.68
10	6 M NaCl	7.7	9.9	6.9	4.2	5.67	5.58	5.90	5.72
11	7 M NaCl	8.6	11.8	....	....	5.68	5.57	5.88	5.69
12	8 M NaCl	9.1	11.8	7.0	5.0	5.68	5.57	5.85	5.69
13	10 M NaCl	10.1	13.8	7.4	6.9	5.71	5.57	5.87	5.72

(a) Before freezing.
(b) After freezing and thawing.
(c) Average pH values for Cows 6 and 7.

permits a separation of meat from expressed juices in a manner which permits a study of pH changes and ionic shifts taking place during processing.

Pronounced changes in meat proteins take place between 40° C. and 70° C. As meat passes through this range pH values are increased.

Between 55° C. and 70° C. some reactions tend to promote protein hydration in such a way as to counteract the effect of denaturation. Dynamic shifts between meat and expressed juice involving sodium, potassium, and calcium ions occur in this critical temperature range. Similar changes involving magnesium ions are not so pronounced.

For studies on drip losses on fresh and thawed frozen meats a temperature of 40° C. may be used. Drip losses on frozen aged meat are much less than on frozen unaged meat. Drip losses on unaged meat are greater for frozen than for fresh meat, whereas the reverse is true for aged meat. Losses decrease with aging both for frozen and unfrozen meat.

Acknowledgments

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Effects of Added Sodium Chloride, Potassium Chloride, Calcium Chloride, Magnesium Chloride, and Citric Acid on Meat Shrinkage at 70° C. and of Added Sodium Chloride on Drip Losses After Freezing and Thawing[a]

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THE WATER HOLDING CAPACITY of meat proteins is directly related to shrinkage on cooking, drip on freezing and thawing, tenderness and perhaps other consumer quality attributes of meat *(2)*.

A previous paper from this laboratory *(6)* points out certain relations between temperature of heating and water holding capacity of meat. Changes in pH and in the amounts of sodium, potassium, calcium, and magnesium retained in the meat and expressed in the juice were observed both with and without added sodium chloride. It seemed appropriate to follow that work with a study of other additives that might affect water holding properties and in turn meat quality. Accordingly, this paper concerns the changes in meat shrinkage at 70° C. when citric acid and the chlorides of sodium, potassium, calcium and magnesium are added. The observation was previously reported *(4)* that sodium chloride infused into meat could greatly decrease drip after freezing and defrosting. Therefore, a more systematic study of this effect is reported also in this paper; there appears to be a direct relation between drip on freezing and thawing and shrinkage on cooking.

METHODS

All methods used for this study were those previously reported *(1, 4, 5, 6)*.

Meat was thoroughly ground and mixed. Meat samples were weighed out and various solutions were added in the ratio of 10 parts of meat to 1 part of solution. When concentrations desired in the meat exceeded those which might be obtained by this simplified procedure, the appropriate amount of salt was added to the meat sample along with 10 ml. of water. The whole was mixed and used after standing a short time.

In view of the results obtained previously, samples were all heated to 70° C. In the case of studies on drip the samples were warmed only to 40° C.

RESULTS AND DISCUSSION

Relations between added sodium chloride, potassium chloride, magnesium chloride, calcium chloride, or citric acid and the shrinkage of meat at 70° C. are shown in Figure 1. Samples of meat were not the same for all curves but the same meat was used for each individual curve. Vertical lines across the curves indicate the approximate limit of taste acceptance of added compounds. These concentrations of additive solution are also noted on each curve. A more critical study of taste acceptance might shift the individual limiting concentrations a bit. The most outstanding finding indicated in Figure 1 is that on meat cooked to 70° C. divalent

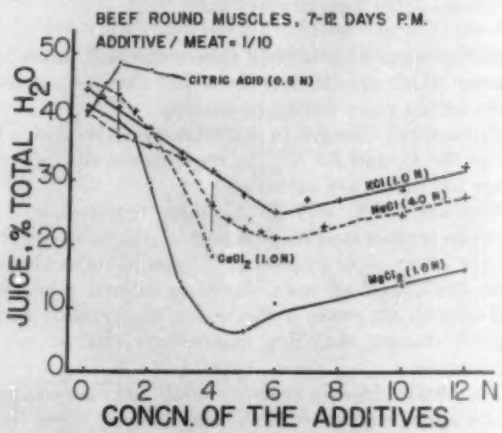

Figure 1. Curves showing the relation between added sodium chloride, potassium chloride, calcium chloride, magnesium chloride or citric acid and the juice expressed by meat heated to 70° C. The concentrations noted in parentheses on each curve indicate the approximate limits of additives from the standpoint of taste.

cations decrease shrinkage to a greater degree than monovalent cations. Citric acid increases shrinkage. To be sure, the maximum effects are far beyond the limits of taste acceptability and yet the striking difference is maintained throughout. It is of interest to compare these findings with those reported previously *(6)*. There, also, quite different results were obtained by comparing the divalent and monovalent ions—the divalent ions tended to go on to the heated meat proteins in 60°-80° C. range. Magnesium and calcium chlorides lower the pH of raw meat (Table 1) and tend to decrease water holding capacity of unheated meat proteins, according to Hamm *(3)*, but apparently this is not so in heated meat where a number of complex reactions are taking place. During heating, pH of meat changes. In the previous report some differences between calcium and magnesium ions were noted. Here, also, it is worthy of note that whereas calcium chloride tended to prevent increase of pH on heating, magnesium chloride did not. A number of suggestions might be given to explain the effects of magnesium and calcium ions on water holding capacity of meat proteins, but for the present such suggestions would be speculative.

[a] This paper reports research supported by the Quartermaster Food and Container Institute for the Armed Forces and is approved for publication. Views and conclusions presented do not necessarily reflect the views or indorsement of the Department of Defense.

[b] Present address: Rath Packing Co., Waterloo, Iowa.

TABLE 1

Maximal amounts of added sodium chloride, potassium chloride, magnesium chloride, calcium chloride, and citric acid with regard to decreasing meat shrinkage and the taste acceptability of the treated ground beef and the effect of these additives on the pH of the fresh and cooked meat (70° C.)

Additive solutions (meat/solution = 10/1)	Effect on max. decrease of shrinkage (a) or taste acceptability (b)	Conc. of the addit. solus. in equivs.	Juice as % total H_2O		pH				Juice	
					Meat before cooking		Meat after cooking			
			Hours after additions							
			½	24	½	24	½	24	½	24
1. No additive[1]	..	....	37.3	.....	5.49	.....	5.81	.....	5.79	.....
2. Dist. H_2O[1] (control)	..	0	44.1	40.7	5.50	5.53	5.83	5.86	5.80	5.78
3. NaCl	a	6.0	24.7	.....	5.57	.....	5.98	.....	5.86	.....
	b	4.0	27.0	25.4	5.53	5.54	5.99	5.90	5.92	5.73
4. KCl	a	6.0	25.2	.....	5.68	.....	6.03	.....	5.89	.....
	b	1.0	38.7	29.2	5.59	5.63	5.90	5.90	5.85	5.88
5. $MgCl_2$	a	4.0	7.4	.....	5.40	.....	5.78	.....	5.67	.....
	b	1.0	39.4	28.5	5.42	5.33	5.77	5.81	5.61	5.65
6. $CaCl_2$	a[2]	4.0	18.1	.....	5.36	.....	5.31	.....	5.05	.....
	b	1.0	43.3	45.8	5.40	5.28	5.59	5.25	5.38	5.16
7. Citric Acid	a[2]	3.0	46.1	.....	4.34	.....	4.46	.....	4.51	.....
	b	0.3	43.6	.....	5.15	.....	5.58	.....	5.48	.....

[1] Average values for the determinations made on 6 different round muscles.
[2] Highest concentrations studied.

Table 1 shows in greater detail pertinent data on adding maximal amounts of the chlorides and citric acid from the points of view of taste and water holding capacity. Contrasting effects of added salts are illustrated together with pH changes on heating and on standing for 24 hours prior to heating to 70° C. The effect of magnesium chloride on improving the water holding capacity of heated meat is clearly evident.

In view of previous work showing that added sodium chloride improved water holding capacity and tenderness of cooked meat *(4, 6)*, it seemed desirable to investigate combinations of sodium, potassium, calcium, and magnesium chlorides. This was particularly of interest since the maximal added amounts from the standpoint of taste were 4 M. NaCl, 1 M. KCl, 0.5 M. $CaCl_2$, and 0.5 M. $MgCl_2$ as 1-10 additive or 2.3% NaCl, 0.75% KCl, 0.6% $CaCl_2$ and 0.5% $MgCl_2$, based on total mixture of meat and added solution. Table 2 shows pH changes and juice expressed on heating to 70° C. for meat which has had combinations of sodium, potassium, calcium, and magnesium chlorides added. Data are shown for meat 0.5 and 24 hours after addition. The hydrating effect of the magnesium chloride is quite clearly shown and the 3.05 M. NaCl, 0.4 M. $MgCl_2$ appeared to be the best. Although an attempt was made to compensate the acidity of the $MgCl_2$ by using a sodium citrate pH 5.6 buffer, no advantage was gained by its use. The alkalizing effect of sodium chloride on meat *(6)* more than compensates for the potential acidity of $MgCl_2$. The action of Versene (disodium salt of ethylene diamine tetracetic acid) is opposite that of the divalent cations and emphasizes the

TABLE 2

Effect of the additions of various salts on the meat shrinkage at 70° C. and on pH

Sample no.	Additive (addit./meat = 1/10)	Juice, % of total moisture lost		pH					
				Hours after the additions					
		½	24	½			24		
				a	b	c	a	b	c
1	Distd. H_2O (d) (control)	44.1	40.7	5.50	5.82	5.83	5.53	5.88	5.82
2	0.1 M Na-Cit. Buffer, pH 5.60 (e)	43.2	42.4	5.48	5.83	5.80	5.50	5.86	5.78
3	0.25 M NaCl soln. (e)	43.0	41.4	5.51	5.84	5.83	5.52	5.90	5.83
4	3.85 M (3.6 + 0.25), NaCl soln. (f)	30.3	28.9	5.45	5.81	5.70	5.54	5.90	5.73
5	3.6 M NaCl/Buffer (g)	33.8	28.8	5.42	5.80	5.69	5.56	5.91	5.76
6	3.05 M NaCl + 0.4 $MgCl_2$ (f)	29.2	23.5	5.35	5.75	5.66	5.40	5.75	5.66
7	2.8 M NaCl + 0.4 M $MgCl_2$/Buffer	31.1	28.4	5.31	5.74	5.64	5.33	5.83	5.63
8	2.25 M NaCl + 0.8 M KCl + 0.4 M $MgCl_2$ (f)	30.0	25.7	5.35	5.83	5.65	5.37	5.87	5.68
9	2.0 M NaCl + 0.8 M KCl + 0.4 M $MgCl_2$/Buffer	32.9	29.2	5.33	5.82	5.69	5.36	5.87	5.66
10	1.0 M KCl soln. (h)	38.7	29.2	5.59	5.90	5.85	5.63	5.90	5.88
11	0.5 M $MgCl_2$ soln. (i)	39.4	28.5	5.40	5.77	5.61	5.33	5.81	5.65
12	1.0 M $MgCl_2$ soln.	29.7	23.5	5.34	5.70	5.55	5.12	5.76	5.61
13	1.0 M $MgCl_2$ + 1.0 M Versene (j)	40.8	37.9	4.62	4.98	4.97	5.31	5.28	5.12
14	0.2 M $CaCl_2$ soln. (k)	40.8	44.3	5.39	5.62	5.50	5.37	5.71	5.51
15	1.0 M $CaCl_2$ soln.	27.4	29.9	5.36	5.51	5.11	5.28	5.25	5.14
16	1.0 M $CaCl_2$ + 1.0 M Versene	41.3	48.5	4.49	4.60	4.60	5.45	5.46	5.47
17	Distd. H_2O (control) (k)	42.8	41.0	5.44	5.71	5.71	5.46	5.94	5.72
18	Distd. H_2O + 1.0 M Versene	39.3	5.04	5.46	5.45	5.16	5.37	5.45	

Legend: (a) pH of the meats before heating; (b) pH of the meats after heating; (c) pH of the juices; (d) Average values for 5 different determinations; (e) Cow 13; (f) Cow 13, 0.25 M. NaCl solution used as solvent for the additives; (g) Cow 13, 0.1 M Na-Citrate bucer pH 5.60, used as solvent for the additive; (h) Cow 8; (i) Cow 9; (j) Disodium salt of ethylenediaminetetraacetic acid dihydrate; (k) Cow 11.

importance of these ions on the water holding capacities of heated meat proteins. When 1 g. Versene alone was added an apparent contradiction arises in that meat shrinkage decreases greatly. However, in this instance all divalent ions are complexed leaving sodium and potassium ions to produce their own hydrating effect. Then, too, there is a possible hydrating effect of the anions of Versene or of the ethylenediamine tetracetic acid itself. The texture of the Versene treated meat was quite different from all other samples of cooked meat.

In order to obtain some insight into the nature of the action of the several chlorides, an extensive study was made of the treated meat with respect to extractability in water and potassium citrate buffer according to a previous report *(5)*. The results indicated no great changes in extractability of the proteins and hence shed little light on the mode of action of the cations.

The decrease in solubility of uncooked meat proteins due to the addition of $MgCl_2$ and the increase in water holding capacity of such proteins on heating presented something of a contrast. To determine the effect of magnesium chloride on tenderness a 19-pound piece of the round of an old cow was taken at 12 days post mortem and stitch pumped to 10% of its weight of a solution containing 2.8 M. sodium chloride and 0.4 M. magnesium chloride. After 24 hours this meat was roasted along with matched control and 29 people unanimously agreed as to its improved eating quality. Twenty-seven rated the treated meat tender and very tender, while 28 persons rated the control tough or very tough. Although part of the effect could be due to the sodium chloride, in view of the data shown in Table 2 and Figure 1, it is believed that magnesium chloride contributed considerably to the improvement of tenderness.

TABLE 3

Effect of sodium chloride on the moisture loss and pH of ground beef heated at 40° C. before freezing and after freezing and thawing

Additive (addit./meat = 1/10)	Juice, % of the total moisture content lost				pH (c)			
	Cow 6		Cow 7		Meat		Juice	
	a	b	a	b	a	b	a	b
No. additive	12.8	28.0	12.7	25.7	5.56	5.55	5.61	5.57
Distd. H_2O (control)	21.2	35.9	19.7	26.6	5.56	5.54	5.59	5.58
1 M NaCl	20.6	30.2	16.2	21.2	5.62	5.55	5.67	5.58
2 M NaCl	15.3	25.6	9.4	10.3	5.64	5.57	5.74	5.60
3 M NaCl	12.8	20.6	7.8	10.3	5.68	5.58	5.81	5.63
4 M NaCl	5.9	14.8	4.2	6.9	5.70	5.55	5.85	5.60
4.5 M NaCl	5.7	13.8	2.2	6.9	5.70	5.54	5.88	5.65
5 M NaCl	5.4	11.3	4.9	6.1	5.67	5.57	5.84	5.70
5.5 M NaCl	6.9	10.8	6.4	4.2	5.74	5.56	5.87	5.68
6 M NaCl	7.7	9.9	6.9	4.2	5.67	5.58	5.90	5.72
7 M NaCl	8.6	11.8	....	....	5.68	5.57	5.88	5.69
8 M NaCl	9.1	11.8	7.0	5.0	5.68	5.57	5.85	5.69
10 M NaCl	10.1	13.8	7.4	6.9	5.71	5.57	5.87	5.72

(a) Before freezing.
(b) After freezing and thawing.
(c) Average pH values for Cows 6 and 7.

Whereas Figure 1 shows the relationship between added sodium chloride and juice expressed on cooking at 70° C., Table 3 shows the relationship between added sodium chloride and drip at 40° C. after freezing and thawing. Data are given also for pH changes in meat and juice (drip). Similarity between the NaCl curve in Figure 1 and the data on drip in Table 3 is striking. It is also worthy of note that the pH shifts toward alkalinity produced by the addition of sodium chloride decreases after freezing and thawing, thus indicating protein modification due to freezing.

SUMMARY AND CONCLUSIONS

The effect of adding citric acid and the chlorides of sodium, potassium, calcium and magnesium to meat has been studied.

Chlorides of sodium, potassium, calcium, and magnesium when added to meat prior to heating increase the water holding capacity of meat proteins when they are heated to 70° C. This holds true except in very high concentrations. The juice expressed on heating was less for the calcium and magnesium chlorides than for the sodium and potassium chlorides. Magnesium chloride showed the most pronounced effect.

Citric acid increased shrinkage when added to meat prior to heating. The additives were also studied in relation to taste acceptance.

Combinations of the above chlorides were studied with sodium and magnesium chlorides in combination showing the greatest effect in promoting water holding capacity of cooked meat. Beef was made more tender by treating with a mixture of these chlorides in solution.

When sodium chloride is added to meat prior to freezing, less drip is encountered on thawing. This relation parallels closely the effect of sodium chloride on juice expressed on cooking to 70° C.

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Thermal Destruction Rates of Thiamine in Pureed Meats and Vegetables[a,b]

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THAT THIAMINE IS HEAT LABILE and that a loss of this nutrient occurs during the heating of foods are well-known facts. During the past 20 years a number of food processing techniques have evolved which show promise of improving the quality of heat processed foods. Many of these are based on achieving sterilization by high-temperature short-time processing or by speeding up the rate of heating (thereby reducing the time required for sterilization) by means of agitation of the containers during processing. With the advent of such processing methods, need for data on their influence on the quality of the product is evident. A review of the literature has indicated that only limited data are available on the stability of thiamine under such conditions. The present investigation was carried on in order to contribute to our knowledge of the kinetics of thiamine destruction in aqueous and buffered solutions and in eight low acid foods over the temperature range of 228° to 300° F.

Watanabe *(33)*, Beadle, Greenwood, and Kraybill *(2)*, and Farrer *(11, 12)* have noted that the thermal destruction of thiamine in aqueous and buffered solutions followed a first order reaction rate. Farrer *(11)* reported that the thermal destruction of thiamine in buffered solution increased with an increase in pH. In order to further evaluate his earlier findings, Farrer *(12)* made a mass action study. He plotted log k (velocity coefficient) against the corresponding pH. The resultant curve showed a change of slope so as to form two straight lines. He concluded that the change in destruction rate was a function of the ionic make-up of the solution and that the velocity coefficient was inversely proportional to the hydrogen-ion concentration. Farrer and Morrison *(15)* reported that the velocity coefficient for thiamine destruction in phosphate buffer solution was related to temperature through the Arrhenius equation in the range of 122° to 230° F. Feaster and Pilcher *(17)* subjected buffered thiamine solutions to heat at temperatures in the range of 180° to 250° F. They concluded that: (a) an increase in pH value from 3.5 to 7.0 caused a progressive decrease in thiamine retention; (b) with a fixed pH value and heating time, thiamine retention decreased with increasing temperature; and (c) at a fixed pH value and temperature, thiamine losses were approximately proportional to time of exposure. Pflug and Esselen *(24)* found that thiamine in M/15 phosphate buffer at pH 7.5 had a destruction rate constant (k) of 2.7 $min.^{-1}$.

Available data on thiamine destruction during canning procedures consist largely of comparisons of the vitamin content of the raw material with that which had been processed. Thiamine losses under such conditions in a number of meats and vegetables have been shown to range from approximately 20 to 70% by a number of workers including Fellers, Esselen, and Fitzgerald *(16)*, Harris *et al. (21)*, Clifcorn and Heberlein *(6)*, Guerrant *et al. (20)*, Lamb, Pressley, and Zuch *(23)*, and Wagner, Strong, and Elvehjem *(32)*. Greenwood *et al. (19)* reported that the thermal destruction of thiamine in pork luncheon meat at temperatures of 210° to 250° F. could be interpreted on the basis of a first order reaction. Rice and Beuk *(25)* studied the rate of destruction of thiamine in pork at temperatures of 120° to 250° F. At temperatures above 170.6° F. the loss of thiamine followed a unimolecular reaction rate.

Weir *(34)* reported that the rate of thiamine destruction in puréed peas increased 2.5 times for each 18° F. rise in temperature in the range of 212° to 298° F. This was found to be true of peas ranging in pH value from 5.1 to 6.5. The author suggested the possibilty that there were two monomolecular reactions involved inasmuch as a change in the rate of reaction occurred after 50% of the thiamine had been destroyed. Bendix *et al. (3)* studied thiamine stability in peas, corn, lima beans, and tomato juice over the temperature range of 220° to 270° F. Only in the case of peas did the thermal destruction data lend itself to interpretation in terms of a first order reaction. The other products showed a decreasing rate of reaction as the exposure time was increased. Feaster, Tompkins, and Ives *(17)* compared the influence of high temperature-short time with conventional processing methods on thiamine retention in peas, carrots, green lima beans, green bean purées, and cream style yellow corn. They showed that the high temperature-short time canning procedure resulted in increased thiamine retention over that obtained with conventional processing times and temperatures. It was further found that increasing the rate of heat penetration through agitation of the cans during processing reduced the amount of thiamine loss by one-third. Similar data on the comparable effects of conventional and short time processing techniques have been reported by Jackson, Feaster, and Pilcher *(22)*, Feaster, Tompkins and Ives *(17)*, Cameron, Pilcher, and Clifcorn *(5)*, and Clifcorn, Peterson, Boyd, and O'Neil *(7)*. A very complete review on the thermal destruction of thiamine in foods has been presented by Farrer *(14)*.

EXPERIMENTAL

Thermal destruction characteristics of thiamine in aqueous and buffered solutions. Studies were made to determine the kinetics of thiamine destruction in Sorenson's phosphate buffer solution of pH 4.5, 5.0, 5.5, 6.0, 6.5, and 7.0, and in aqueous solution at pH 3.5. Destruction rates were determined at temperatures of 228°, 246°, 264°, 282°, and 300° F.

Preparation of samples. A stock thiamine solution was prepared by dissolving 200 mg. of U.S.P crystalline thiamine

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chloride hydrochloride in 25% ethyl alcohol and bringing to volume in a 100 ml. volumetric flask. One ml. of the stock thiamine solution was diluted to 500 ml. with the different buffer solutions, and the pH was checked using a Beckman Model G pH meter. An aqueous thiamine solution was prepared by diluting one ml. of the stock thiamine solution to 500 ml. with distilled and demineralized water. The pH value of the aqueous thiamine solution was 3.5. Two ml. portions of these thiamine solutions were filled into Pyrex thermal death time tubes [Esty and Williams, *(10)*]. The tubes were sealed in an oxy-gas flame and stored at 0° F. until such time as they were used in the heating tests. Final thiamine concentration of the samples was 4 μg. per ml. This level was selected as being representative of the thiamine content of foods. Preliminary tests indicated that the rate of destruction of thiamine in M/15 phosphate buffer was not influenced by its concentration in the range of 1 to 20 μg. per ml.

Apparatus for heating samples. Samples were heated in a miniature retort of a type similar to that described by Schmidt *(27)*. It could be heated up to temperature and cooled very rapidly. It was equipped with automatic controllers so that the temperature could be regulated to within ± 0.25° F. In each run an extra tube of sample equipped with a 30 gauge B.S. copper-constantan thermocouple at the cold zone was included so that heating and cooling rates could be recorded. The thermocouple was connected with a recording potentiometer which recorded the temperature at 15 second intervals. The heating and cooling lag correction factor for the tubes under the experimental conditions was determined by essentially the same procedure described by Sognefest and Benjamin *(29)*. The lag correction factor was found to be 0.6 minutes and was taken into consideration in the calculated heating times.

Treatment of samples. Groups of 3 tubes of each sample were heated for each time and temperature interval. Three control samples were carried through the entire procedure, except for heating, when each thermal destruction test was made. This procedure made it possible to calculate and express results in terms of percent thiamine remaining in the samples after a given heat treatment. Samples were stored at 0° F. until needed for analysis.

Analysis of samples. One ml. of sample was diluted to 25 ml. with acidified 25% KCl. Five ml. aliquots were taken for analysis in accordance with the method of the Association of Vitamin Chemists *(1)*. Because the analyses were being made on pure solutions, the extraction and purification steps were omitted. Fluorescence was measured with a Model 12-B Coleman Electronic Photofluorometer, using quinine sulfate (300 μg. per ml.) as a standard.

Treatment of data. The order of a destruction rate reaction may be determined graphically by plotting different functions of concentrations, or percent retention, against time. A first order reaction is characterized by a straight line when the log of the concentration of the substance is plotted against time. This plot yields a rate of destruction curve. The slope of this curve, designated by the letter D, represents the time required to destroy 90% of the material originally present. (D is defined as the number of minutes traversed by the curve in passing through one log cycle.) From the D values obtained at different temperatures it is possible to derive a thermal destruction time (TDT) curve. A curve of this type, which is obtained by plotting the log of the D value against the corresponding temperature, defines the thermal destruction characteristics at a given temperature as well as the sensitivity of the material to changes in temperature.

Interpretation of results. Thiamine retention was expressed in terms of the percentage remaining after a given heat treatment. Retention values for three replicates, at each heating time, were used to calculate rate of destruction regression equations, using the procedure outlined by Bliss *(4)*. D values were obtained by finding the reciprocal of the slope, or b, from the regression equations. Thermal destruction time (TDT) curves were derived by plotting the log of D against the corresponding temperature.

The velocity coefficient, k, expressed in reciprocal time units, was determined graphically from the D value through use of the equation:

$$k = \frac{2.30}{D}$$

This equation was derived from the differential equation: $-(dc/C)/dt = k$, where $-(dc/C)$ represents the fractional decrease in concentration C, occurring during the time dt. The integrated form of the equation is based on the 90% level of destruction, as indicated by the use of the D value.

The half-life, $T_{1/2}$, was calculated by use of the relationship: $T_{1/2} = \frac{.693}{k}$. This value represents the time required to destroy 50% of the thiamine originally present.

The relationship between thiamine destruction and temperature was obtained from a plot of the log of the velocity coefficient, k, against the reciprocal of the absolute temperature. A resultant straight line is taken to indicate that the reaction satisfied the Arrhenius equation:

$$\log k = \log A - \frac{E}{2.303\,RT}$$

in which A is the frequency factor, R is the gas constant, and E is the energy of activation. The constant E, which is characteristic of the reaction and determines the influence of temperature on the reaction rate, was calculated from the slope of the curve obtained by plotting log k against 1/T.

As a means of comparison with data concerned with the destruction of bacterial spores, the temperature coefficient, Q_{10}, was calculated from the relationship:

$$Q_{10} = \frac{k_{(t)}}{k_{(t-10)}}$$

in which $k_{(t)}$ is the reaction rate constant at temperature T_1, and $k_{(t-10)}$ is the reaction rate constant at a temperature 18° F. (10° C.) below T_1.

Thermal destruction of thiamine in pureed meats and vegetables. *Preparation of Puréed Foods:* Green bean, pea, and spinach purées were prepared from commercially frozen products. Carrrot purée was prepared using fresh carrots which were washed, skinned, cut into chips approximately one-half inch thick, and blanched for 3 minutes in boiling water. The vegetables were passed, without prior thawing, through a Fitzpatrick Comminuting Machine [4] fitted with a number A-4 screen. The resulting coarsely ground material was then passed successively through screens of finer mesh (numbers A-2 and C-40).

Meats were trimmed to remove excess fat and connective tissue. They were then frozen at 0° F. to facilitate handling during the following steps. The blocks of frozen meat were cut into two-inch squares with a meat saw. These pieces of meat were then ground twice in a food chopper with a coarse screen. The products were then passed through the comminuting machine fitted with a number A-2 screen.

Moisture determinations were made on each product, and water was added as required to adjust them to moisture levels approximating those of commercially prepared puréed foods. Final moisture content and pH value of these products are shown in Table 1. Products were packed in 20-ounce containers and stored at 0° F.

Heating Samples: The samples were heated in metal thermal death time (TDT) cans.* This container, designated as 208 by 006 (i.e. 2½ inch diameter and ⅜ inch depth), has rapid heating and cooling rates. Heat penetration tests on each of the products showed that the samples could be heated to retort temperature (RT) in 1.75 to 2.0 minutes.

The containers of product were removed from freezer storage and the contents thawed by holding in flowing water at approximately 150° F. Contents were mixed in a Waring blender and 14-g. portions were weighed into clean, enamel lined T.D.T. cans. The containers were then sealed under vacuum (22-23

[4] Manufactured by W. J. Fitzpatrick Co., Chicago, Illinois.

TABLE 1

Moisture content, total and free thiamine content, and initial pH value of products used in study of thermal destruction of thiamine.

Product	Total thiamine content	Free thiamine content	Moisture content	pH
	mg./100 g.	%	%	
Group A[1]				
Carrots	.096	54	90.7	6.13
Green beans	.124	56	93.2	5.83
Peas	.430	81	84.4	6.75
Pork	.680	87	77.2	6.18
Group B[1]				
Spinach	.132	24	93.5	6.70
Beef heart	.420	42	82.7	6.10
Beef liver	.235	45	79.2	6.07
Lamb	.213	12	80.3	6.18

[1] Grouping based on analysis of variance of thiamine destruction rate.

inches) on a Pacific No. 1 Semi-Automatic Vacuum Sealing Machine.* The filled and sealed TDT cans were then held frozen at 0° F. until required for heating tests. Just prior to use the cans were held at room temperature for one hour to thaw the contents.

Four TDT cans of a given product were exposed at each time and temperature interval. One can of each group was fitted with a copper-constantan (30 gauge B.S.) thermocouple which was located in the zone of slowest heating. The cans were heated in the miniature retort and by the same procedure as described previously in the study of aqueous and buffered solutions of thiamine. The temperature of the zone of slowest heating was followed through the use of a Brown Strip Chart Multiple Point Electronik Potentiometer, which recorded the temperature at 15-second intervals. After heating, cans were stored at

* Supplied through the courtesy of the American Can Co., Maywood, Ill.

TABLE 2

Thiamine destruction rates in M/15 phosphate buffer and distilled water.

Temperature									
228° F.		246° F.		264° F.		282° F.		300° F.	
Heating time	Thiamine retention[1]	Heating time	Thiamine retention[1]	Heating time	Thiamine retention[1]	Heating time	Thiamine retention[1]	Heating time	Thiamine retention[1]
min.	%	*min.*	%	*min.*	%	*min.*	%	*min.*	%
Buffer—pH 4.53									
0	100	0	100	0	100	0	100	0	100
20.9	94	51.9	92	8.2	88	5.9	78	2.9	76
60.9	85	30.9	84	10.7	85	8.4	74	3.7	71
120.9	73	45.9	78	20.9	72	10.9	66	5.9	59
150.9	70	60.9	70	40.7	54	20.7	46	8.4	47
280.9	51	100.9	56	60.9	42	41.2	22	10.9	38
Buffer—pH 5.00									
0	100	0	100	0	100	0	100	0	100
20.9	93	10.9	93	5.7	91	3.9	85	2.9	75
60.9	85	30.9	81	8.2	87	5.9	78	3.7	70
120.9	71	45.9	74	10.9	83	8.4	70	5.9	56
150.9	66	60.9	66	20.9	71	10.9	64	8.4	45
280.9	44	100.9	52	40.7	51	20.7	44	10.9	36
Buffer—pH 5.50									
0	100	0	100	0	100	0	100	0	100
30.9	91	10.9	93	4.2	92	2.6	88	1.9	78
90.9	75	30.9	79	5.7	90	3.9	83	2.9	68
120.9	67	45.9	69	8.2	86	5.9	73	3.7	61
150.9	59	60.9	62	10.9	81	8.4	66	5.9	47
280.9	36	100.9	47	20.9	67	10.9	55	10.9	26
Buffer—pH 6.00									
0	100	0	100	0	100	0	100	0	100
20.9	89	5.9	91	2.4	93	1.9	84	1.2	78
60.9	68	15.9	80	4.2	87	2.6	81	1.9	66
90.9	60	30.9	67	5.7	83	3.9	72	2.9	54
120.9	50	45.9	56	8.2	77	5.9	60	3.7	44
150.9	43	80.9	46	10.7	71	8.4	50	5.9	28
Buffer—pH 6.50									
0	100	0	100	0	100	0	100	0	100
5.9	90	3.9	87	1.4	86	1.4	69	0.9	54
20.9	73	5.9	83	2.4	79	1.9	61	1.2	42
30.9	65	10.9	64	4.2	69	2.6	50	1.9	28
60.9	44	15.9	58	5.7	59	3.9	38	2.9	12
90.9	26	30.9	37	8.2	48	5.9	20	3.7	7
Buffer—pH 7.00									
0	100	0	100	0	100	0	100	....	....
2.9	85	1.2	81	1.2	62	0.9	34	....	....
5.9	73	1.9	75	1.4	54	1.2	31	....	....
7.9	67	2.9	65	2.4	42	1.4	26	....	....
10.9	52	3.9	57	4.2	23	1.9	18	....	....
20.9	37	4.9	46	5.7	12	1.9	17	....	....
Distilled Water—pH 3.50									
0	100	0	100	0	100	0	100	0	100
20.9	92	15.9	91	20.9	73	5.9	81	2.9	79
60.9	80	30.9	85	40.7	54	10.9	62	3.7	72
120.9	72	45.9	80	60.9	38	20.7	44	5.9	62
150.9	70	60.9	71	90.9	23	41.2	23	10.9	38
280.9	47	100.9	53	120.9	16	60.9	6	20.9	14

[1] Mean retention based on three replicates showing agreement within ± 3 percent.

0° F. until the thiamine content of their contents could be determined.

Selection of Heating Times: Heating and cooling rate data were obtained for each product in order to calculate approximate heating lag correction factors.

The thermal destruction of thiamine was found to follow a first order reaction rate and hence the initial concentration of the vitamin is without influence on the characteristics of the reaction. Therefore, the small losses encountered during the heating and cooling periods do not affect the nature of the thermal breakdown. It was decided to adopt a procedure whereby a set of samples would be heated for 2 minutes, this being the longest come-up time encountered in the product studies. These samples were then taken to represent the 100% thiamine retention level. Each of the samples heated at subsequent times would also receive the same treatment accorded the control samples.

Advantages of using such a procedure were pointed out by Roseveare *(26)*, and Daniels *(8)* who showed that inaccuracy may result from using data obtained at the very beginning of a reaction. An additional advantage results from the fact that, using this procedure, the amount of thiamine destroyed during each heating period is a measure of actual exposure at the temperature of study, and not at temperatures encompassed during the heating and cooling phases of the process. The final adoption of the procedure was predicated on the fact that extremely good reproducibility of heating and cooling data could be obtained.

Analysis of Samples: Previously heated TDT cans were opened prior to thawing (after storage at 0° F.) so as to eliminate sample loss. Contents of each can were thoroughly macerated with a mortar and pestle, and a 10-g. sample was taken for analysis of thiamine in accordance with the procedure outlined by the Association of Vitamin Chemists *(1)*. The pH value of each sample was also determined using a Beckman Model G pH Meter.

Treatment of data. The experimental data were subjected to the same calculations as described previously in the case of studies on thiamine destruction in aqueous and buffered solutions.

RESULTS AND DISCUSSION

Thiamine destruction in aqueous and buffered solutions. Table 2 summarizes the thermal destruction rates of thiamine in aqueous and buffered solutions at temperatures of 228°, 264°, 282° and 300° F. These data represent the mean percent retention of thiamine for 3 replicate samples in each case. Variation in percent thiamine retention among the 3 replicates never exceeded 3%. The D, k, and T½ values as derived from regression equations for each pH at the 5 temperatures are shown in Table 3.

Thermal destruction time (TDT) curves for thiamine at different pH values are presented in Figure 1. The slopes of these curves, or z values, are in the range of 44° to 46° F., with a mean of 45° F. Examination of the curves indicates that rate of increase in thiamine destruction is approximately doubled by an 18° F. rise in temperature. Calculated Q_{10} value for thiamine was 2.5.

In attempting to evaluate the influence of pH on the destruction rate, two facts were noted. Reference to the TDT curves in Figure 1 shows that the slopes are closely similar for all of the pH levels studied. The pH change caused a displacement of the curve along the time axis in such a manner as to indicate that an increase in the pH value caused the destruction rate to increase. A more significant result of increase in pH may be seen in Figure 2, which comprises a plot of the log of the velocity coefficient, k, against the pH of the solutions. In attempting to draw a curve through the points, it was found that the data lent itself more readily to two separate lines. Farrer *(12)* obtained

TABLE 3

D values (time for 90 percent destruction), velocity coefficients (k), and half-life values (T½) for thiamine destruction in aqueous and buffered solution in the temperature range 228° to 300° F.

pH	Temperature				
	228° F.	246° F.	264° F.	282° F.	300° F.
	D Value (minutes)				
4.53	952.4	398.3	159.2	63.4	25.8
5.00	790.5	349.3	137.7	58.5	24.4
5.50	630.0	306.5	122.8	43.6	18.6
6.00	413.2	182.5	70.8	28.6	10.5
6.50	159.7	70.2	26.4	8.7	3.2
7.00	48.9	15.3	6.6	2.8	1.1[1]
3.50 (Aqueous)	900.2	373.6	147.7	51.4	24.4
	Velocity coefficient (minutes^{-1})				
4.53	.0024	.0058	.0145	.0363	.0893
5.00	.0029	.0066	.0167	.0394	.0944
5.50	.0037	.0076	.0188	.0528	.1240
6.00	.0056	.0126	.0325	.0805	.2190
6.50	.0144	.0328	.0872	.2660	.7220
7.00	.0471	.1510	.3500	.8280	2.0900[1]
3.50 (Aqueous)	.0026	.0062	.0156	.0448	.0944
	Half-life Value T½ (minutes)				
4.53	286.3	120.0	47.8	19.1	7.8
5.00	238.2	105.3	41.5	17.6	7.4
5.50	190.0	92.3	36.8	13.1	5.6
6.00	124.3	55.0	21.4	8.6	3.2
6.50	48.2	21.2	8.0	2.6	1.0
7.00	14.7	4.6	2.0	0.8	0.3[1]
3.50 (Aqueous)	271.0	112.5	44.4	15.5	7.3

[1] From extrapolated data.

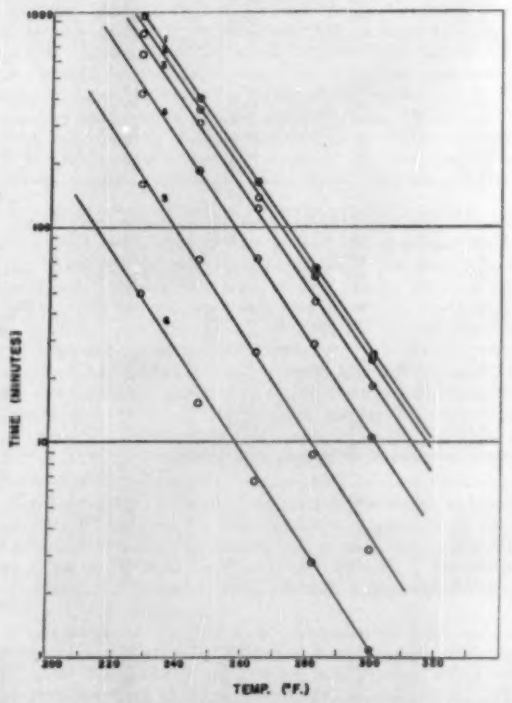

Figure 1. Thermal destruction time (time for 90% destruction) curves for thiamine in phosphate buffer solutions. Curve 1, pH 4.53; curve 2, pH 5.0; curve 3, pH 5.5; curve 4, pH 6.0; curve 5, pH 6.5; curve 6, pH 7.0.

TABLE 4

Thermal destruction rates of thiamine in strained carrots, green beans, peas, spinach, beef heart, beef liver, lamb and pork at 228°, 246°, 264°, 282° and 300° F.

Temperature														
228° F.			246° F.			264° F.			282° F.			300° F.		
Time	pH	Thiamine Retention	Time	pH	Thiamine Retention	Time	pH	Thiamine Retention	Time	pH	Thiamine Retention	Time	pH	Thiamine Retention
Min.		%	*Min.*		%	*Min.*		%	*Min.*		%	*Min.*		%
Carrots														
0	6.05	100	0	5.95	100	0	5.87	100	0	5.80	100	0	5.72	100
30	5.45	82	5	5.61	93	6	5.55	80	2	5.49	86	1	5.54	87
60	5.25	73	10	5.52	88	12	5.40	68	5	5.35	72	2	5.34	72
80	5.08	65	20	5.28	78	23	5.19	50	9	5.17	50	4	5.19	50
150	4.95	48	40	5.08	61	40	4.97	33	14	5.03	38	6	5.11	38
240	4.80	30	70	4.90	43	60	4.88	17	20	4.90	25	8	4.97	27
Green Beans														
0	5.82	100	0	5.82	100	0	5.77	100	0	5.76	100	0	5.72	100
30	5.70	83	5	5.68	93	6	5.58	79	2	5.67	86	1	5.58	85
60	5.60	74	10	5.68	88	12	5.42	68	5	5.50	71	2	5.42	65
80	5.56	63	20	5.52	77	23	5.32	48	9	5.35	51	4	5.28	46
150	5.40	47	40	5.42	63	40	5.12	29	14	5.23	40	6	5.18	35
240	5.28	30	70	5.22	43	60	5.02	15	20	5.15	23	8	5.10	25
Peas														
0	6.68	100	0	6.71	100	0	6.62	100	0	6.59	100	0	6.56	100
30	6.48	81	5	6.60	92	6	6.48	81	2	6.45	87	1	6.45	92
60	6.28	69	10	6.50	88	12	6.32	70	5	6.31	71	2	6.33	72
80	6.21	64	20	6.36	80	23	6.12	52	9	6.20	53	4	6.18	53
150	6.09	46	40	6.19	62	40	5.98	35	14	6.07	39	6	6.13	38
240	5.92	29	70	6.03	45	60	5.85	18	20	5.95	24	8	5.98	28
Spinach														
0	6.67	100	0	6.60	100	0	6.43	100	0	6.40	100	0	6.23	100
30	6.28	76	5	6.45	90	6	6.04	76	2	6.15	86	1	6.00	73
60	5.95	60	10	6.28	81	12	5.87	61	5	5.90	65	2	5.84	60
80	5.82	52	20	6.03	67	23	5.60	42	9	5.63	49	4	5.58	40
150	5.59	30	40	5.82	52	40	5.48	27	14	5.42	32	6	5.50	24
240	5.48	20	70	5.65	36	60	5.29	12	20	5.33	19	8	5.38	16
Beef Heart														
0	6.10	100	0	6.08	100	0	6.09	100	0	6.10	100	0	6.10	100
30	6.10	73	5	6.09	90	6	6.08	71	2	6.10	89	1	6.11	77
60	6.09	60	10	6.09	85	12	6.10	50	5	6.09	60	2	6.10	60
80	6.08	53	20	6.10	73	23	6.10	36	9	6.08	41	4	6.09	40
150	6.09	32	40	6.08	53	40	6.10	18	14	6.10	25	6	6.10	28
240	6.09	18	70	6.08	33	60	6.09	9	20	6.09	12	8	6.08	17
Beef Liver														
0	6.08	100	0	6.07	100	0	6.06	100	0	6.05	100	0	6.06	100
30	6.02	78	5	6.05	91	6	6.02	84	2	6.05	86	1	6.04	76
60	6.00	65	10	6.01	86	12	6.00	65	5	6.02	65	2	6.03	60
80	5.96	60	20	5.95	73	23	5.95	44	9	6.00	47	4	5.95	41
150	5.94	38	40	5.87	55	40	5.88	26	14	5.91	29	6	5.80	25
240	5.90	20	70	5.64	33	60	5.60	11	20	5.80	17	8	5.62	15
Lamb														
0	6.18	100	0	6.18	100	0	6.17	100	0	6.18	100	0	6.18	100
30	6.18	81	5	6.17	90	6	6.16	75	2	6.18	86	1	6.18	88
60	6.18	67	10	6.18	83	12	6.17	55	5	6.19	64	2	6.17	74
80	6.10	60	20	6.19	76	23	6.18	36	9	6.18	49	4	6.17	44
150	6.19	37	40	6.17	56	40	6.17	20	14	6.17	31	6	6.18	32
240	6.19	23	70	6.16	37	60	6.15	10	20	6.16	20	8	6.16	23
Pork														
0	6.18	100	0	6.18	100	0	6.18	100	0	6.18	100	0	6.18	100
30	6.18	84	5	6.17	91	6	6.17	83	2	6.18	88	1	6.18	83
60	6.17	70	10	6.17	88	12	6.18	70	5	6.18	71	2	6.17	70
80	6.18	62	20	6.18	79	23	6.18	50	9	6.18	51	4	6.18	50
150	6.17	43	40	6.17	61	40	6.17	30	14	6.16	37	6	6.17	36
240	6.16	26	70	6.15	40	60	6.15	18	20	6.16	24	8	6.16	26

similar results in a study at 212° F. and attempted to explain the matter in terms of a change in the ionic constitution of the buffer medium. From Figure 2 it may be seen that the break in the log k/pH curve occurs between pH 6.0 and 6.5. The D values (Table 3) also indicate that the most pronounced change occurred between pH 6.0 and 6.5. When the log of the velocity coefficient, k, was plotted against the reciprocal of the absolute temperature the straight lines obtained showed that the reaction involved in the cleavage of the thiamine molecule by heat satisfies the Arrhenius equation. The slopes of these lines indicated that the energy of activation, E, for the thiamine destruction is 28.8 k calories per mole.

Thiamine destruction in pureéd meats and vegetables. Data on the mean thiamine retention in puréed meats and vegetables are summarized in Table 4. Each retention value represents the mean of 3 replicates agreeing within ± 5%. The D values, velocity coefficients (k), and half-life values ($T_{1/2}$) are shown in Table 5. An examination of these data suggests a difference in the rates of thiamine destruction in the

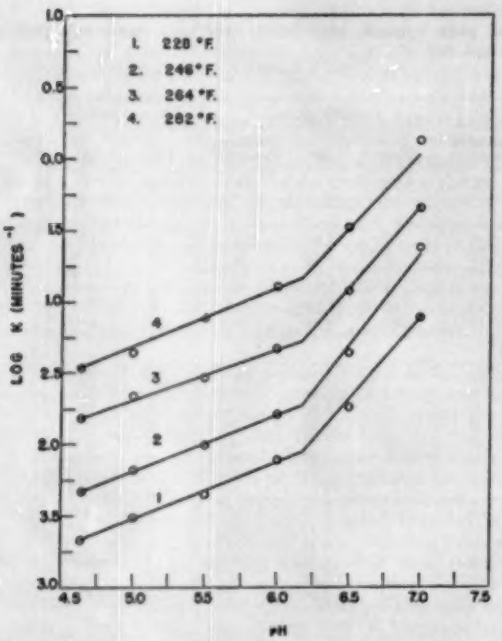

Figure 2. Curves representing plot of pH versus the log of the velocity coefficients for the destruction of thiamine in phosphate buffer solutions.

eight products studied. In order to evaluate these differences, a statistical analysis of variance was carried out according to the method of Snedecor *(28)* for data with two criteria of classification. The null hypothesis tested was that all the products are random samples from the same population. Results of the analysis of variance, based on the D values, are presented in Table 6. Since the calculated F value was greater than the $F_{.05}$, the null hypothesis was rejected.

Rejection of the null hypothesis indicated that either: (a) the eight products each constituted a separate population; or (b) some of the products belong to one population and some to another. Data in Table 6 were then subjected to Tukey's *(31)* test of significance of difference between any two means in an analysis of variance. Tukey's test of significance of difference value (T_s) was calculated from the equation:

$$T_s = (q \sqrt{1/r}) (\sqrt{S^2})$$

where q is the range of k treatments divided by the standard deviation. The value may be obtained from tables presented by Dixon and Massey *(9)*.

r is the number of replicates.

S^2 is the mean square of the experimental error.

In the present case the q value was found to be 4.67, r is 5, and S^2 is 540.0. Substituting in the equation:

$$T_s = (4.67 - \sqrt{1/5}) (\sqrt{540}) = 95.70$$

Therefore, any sums of D values varying by as much as 95.7 are from a different population. The results (Table 7) indicate that the products may be divided into two groups as follows: (1) carrots, green beans, peas, and pork; and (2) spinach, beef heart, liver, and lamb.

These two groupings were checked by subjecting each to an analysis of variance to test the null hypothesis that the data constituted random samples from the same population. It was found that the null hypothesis could be retained and that it could be said that, under the experimental conditions employed, the products in each of the two groups showed no significant difference.

TABLE 5

D values (time for 90 percent destruction), velocity coefficients (k), and half-life value ($T_{½}$) for thiamine destruction in strained vegetables and meats at 228°, 246°, 264°, and 300° F.

Product	Temperature				
	228° F.	246° F.	264° F.	282° F.	300° F.
D Value (minutes)					
Carrots	471.7	191.6	80.7	32.4	13.8
Green beans	465.6	188.7	74.1	32.1	13.2
Peas	454.5	202.3	83.3	32.5	13.9
Spinach	344.8	161.3	68.5	27.9	10.1
Beef heart	338.1	146.6	58.7	21.7	10.6
Beef liver	342.5	157.1	63.3	25.8	9.9
Lamb	370.3	166.7	61.0	28.3	11.9
Pork	416.7	178.6	80.0	32.1	13.6
Velocity Coefficient k (minutes⁻¹)					
Carrots	.0049	.0120	.0285	.0711	.1669
Green beans	.0049	.0122	.0311	.0717	.1744
Peas	.0051	.0114	.0276	.0708	.1657
Spinach	.0067	.0143	.0336	.0825	.2280
Beef heart	.0068	.0157	.0392	.0161	.2172
Beef liver	.0067	.0147	.0364	.0892	.2326
Lamb	.0062	.0138	.0377	.0814	.1935
Pork	.0055	.0129	.0288	.0717	.1693
Half-life Value $T_{½}$ (minutes)					
Carrots	142.0	57.8	24.3	9.7	4.2
Green beans	140.0	56.8	22.3	9.7	4.0
Peas	136.7	60.8	25.1	9.8	4.2
Spinach	103.8	48.5	20.6	8.4	3.0
Beef heart	101.8	44.2	17.7	6.5	3.2
Beef liver	103.2	47.2	19.0	7.8	3.0
Lamb	111.5	50.2	18.4	8.5	3.6
Pork	125.4	53.7	24.1	9.7	4.1

Thermal destruction time curves for thiamine in the above two groups of products are shown in Figure 3. The z values for thiamine destruction in these products lie in the range of 45° to 47° F. and are of the same magnitude as was found in the case of aqueous and buffered solutions.

On the basis of the types of products in each group there did not appear to be any obvious reason for the differentiation. Farrer *(13)* studied thiamine destruction in potatoes, carrots, peas and cabbage heated at 212° F. and reported that there was little or no difference in the destruction rate.

TABLE 6

Analysis of variance of D values representing the rate of destruction of thiamine in low acid foods heated at temperatures in the range of 228° to 300° F.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Total	39	8,462.40	
Temperatures	4	8,196.50	
Products	7	114.68	16.38
Discrepancy	28	151.20	5.40

Test for Products ..... $F = \frac{16.38}{5.4} = 3.04^1$

[1] $F_{.05}$ for 7 and 28 d.f. = 2.36; $F_{.01}$ for 7 and 28 d.f. = 3.36; F values from Snedecor *(28)*.

In a consideration of the relationship of the pH value of the different foods studied to thiamine destruction rates, it is apparent that the hydrogen ion concentration was not the sole factor involved. Also, variations in the proximate composition of the foods do not appear to be directly related to thiamine stability. Another possibility for the variation in thiamine destruction rate was thought to be related to the fact that the products varied in their content of free and combined thiamine.

TABLE 7

Sums of D values for each product for use in checking the Tukey (31) test of significance [1]

Product	Sum of D values	Ts. difference between sum of D values of product and that of largest D sum
Carrots	790.2	.....
Peas	786.5	3.7
Green Beans	773.7	16.5
Pork	721.0	69.2
Lamb	638.2	152.0[2]
Spinach	612.6	177.6[2]
Beef Liver	598.6	191.6[2]
Beef Heart	575.7	214.5[2]

[1] Tukey T_5 = 95.7 (as calculated).
[2] Indicates that the samples are significantly different at the 5% level.

Tauber *(30)* reported that vegetables contain only a small proportion of their vitamin B_1, in the combined form. Analyses were made to determine the relative amounts of free and combined thiamine in each of the eight products. Results are shown in Table 1. These data indicate that vegetables may differ. Of the four vegetables, only peas showed a lesser proportion of combined thiamine, whereas spinach contained three times as much combined as free thiamine.

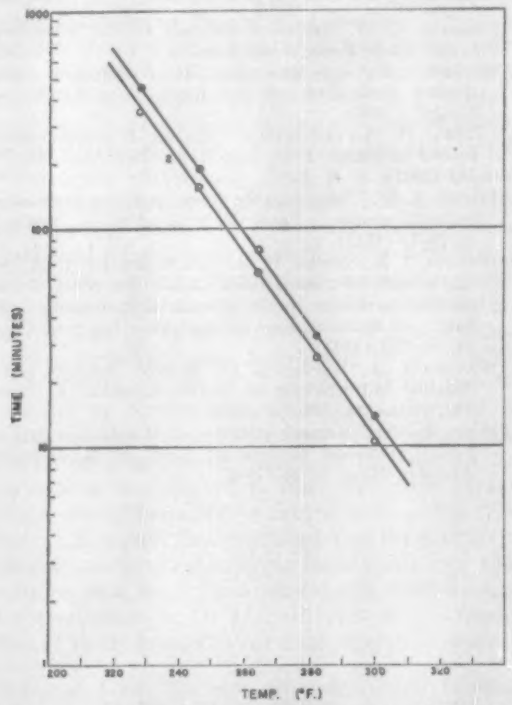

Figure 3. Thermal destruction time (time for 90% destruction) curves for thiamine in strained foods. Curve 1, average curve for carrots, green beans, peas and spinach; curve 2, average curve for beef heart, beef liver, lamb and spinach.

It is difficult to account for the variation in thiamine destruction rates solely on the basis of the differences in the proportions of the free and combined forms of thiamine. It is possible that the variation in thiamine destruction is dependent on an interrelationship between hydrogen ion concentration and free thiamine content. Products having a lower relative hydrogen ion concentration are able to counteract the higher relative proportion of combined thiamine, which would tend to increase the destruction rate. This relationship may be seen in the case of carrots and green beans which, although they contained 46 and 44% combined thiamine, respectively, had pH values of 5.92 and 6.12. These products showed thiamine destruction rates similar to that of peas which contained only 19% combined thiamine and had a pH value of 6.75. In direct contrast, spinach not only had a higher combined thiamine content (76%) but also a higher pH value. These relationships might account for the fact that thiamine in spinach showed a more rapid rate of thermal destruction.

In the case of meat products, the variation in hydrogen ion concentration was less than that of vegetables. It would seem, therefore, that the difference in thiamine destruction rates may be more directly associated with the concentration of combined thiamine. Pork contained 87% free thiamine and showed slower destruction characteristics than did the other meats which contained smaller proportions of the free thiamine.

In general, the rate of destruction of thiamine in foods could be related to two factors, pH and combined thiamine content. The relationship might indicate that high pH and high combined thiamine content increase the rate of destruction of this vitamin.

When the log of the velocity coefficient, k, of the 8 foods was plotted against the reciprocal of the absolute temperature, T, the resultant straight lines indicated that the thermal destruction of thiamine satisfied the Arrhenius equation. The energy of activation for the reaction involved in the breakdown of the thiamine molecule was calculated to be 27.0 k cal.

SUMMARY AND CONCLUSIONS

The rate of destruction of thiamine in aqueous solution, phosphate buffered solutions and selected low-acid foods was studied over the temperature range of 228° to 300° F. The reaction involved in the thermal cleavage of the thiamine molecule was found to be of the first order type through the entire temperature range studied. The energy of activation for the breakdown of thiamine in the different media was in the range of 27 to 28.8 k cal. per mole.

The rate of thiamine destruction in phosphate buffered solutions in the pH range of 4.5 to 7.0 increased with increasing pH. The most pronounced change in the reaction rate occurred between pH 6.0 and 6.5. The Q_{10} value for thiamine destruction was approximately 2.5, irrespective of pH or composition of the material substrate.

Thiamine in foods is more resistant to thermal breakdown than is the pure vitamin in aqueous or buffered

solutions. The variation is independent of differences in pH since solutions of similar pH to that of specific foods showed more rapid destruction characteristics. It is suggested that the thermal destruction of thiamine in foods may be dependent on the interrelationship of pH and the relative proportions of the free and combined forms of the vitamin. At a given pH level the rate of destruction of thiamine increased with increasing content of the combined form.

Carrots, green beans, peas, and pork showed similar thermal destruction characteristics for thiamine. The thermal destruction of thiamine in spinach, beef heart, beef liver, and lamb was more rapid than in the former four products.

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Role of Reducing Sugars and Amino Acids In the Browning of Potato Chips[a, b]

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COLOR IS ONE OF THE MOST IMPORTANT ATTRIBUTES of quality for potato chips. There is also a relationship between color and flavor of potato chips. The pigments which develop when potatoes are fried determine the color of the chips and impart a noticeable "caramel-like" flavor. Browning of potato chips has been generally attributed to the "caramelization" of reducing sugars at the relatively high temperatures used for frying. Several authors have shown a highly significant correlation between the reducing sugar content of potato tubers and the color of the chips produced therefrom *(19, 14, 21, 26)*. Recently, however, Legault, Talbert, Mylne and Bryan *(10)* indicated that browning of chips is due, in part at least, to a Maillard type of reaction between reducing sugars and amino acids. Patton and Pyke *(12)* also presented evidence to indicate that the Maillard reaction is responsible for the color of potato chips. Shallenberger *(15)* found that "the effect of nitrogenous substances on the development of color in potato chips appeared to limit the development of color in the chips as opposed to the catalytic effect nitrogen has on caramelization." More recently Habib and Brown *(7)* proved a correlation between total solids, reducing sugars, and free amino nitrogen contents of raw potatoes and the color of the chips. They concluded that, although reducing sugars are the most important factor determining the color of the chips, their importance in some cases may be limited by the relative amount of free amino nitrogen and hence both factors must be considered when evaluating any variety for chipping. There is a large volume of literature concerning the relative reactivity of various types of sugars and amino acids. Apparently there is a great similarity between the reaction of aldoses and ketoses insofar as the browning reaction is concerned, but some factors might tend to retard the reaction of one type of sugar or the other. It has generally been agreed that pentoses react more rapidly than hexoses, which in turn are more reactive than di- or tri-saccharides. It is also believed that non-reducing sugars brown only under conditions which involve hydrolysis *(2, 4, 21)*. It has also been indicated by several research workers that reducing sugars and the free amino groups of proteins combine initially in a one-to-one ratio. In this respect basic amino acids are believed to react with more carbonyl groups than either acidic or neutral amino acids. Some workers, however, have indicated that the ratio of the combination between reducing sugars and free amino groups approaches 1.5 to 1 ratio during the later stages of the reaction *(6, 11, 13)*. In view of the catalytic effect of amino groups on reducing sugars; i.e., transformation without apparent sugar-amine condensation, it was found that mole for mole diamino acids were more effective than either neutral or dicarboxylic acids *(18, 20, 24)*. Few data were found relating to the effect of the various types of sugars and amino acids in potato tubers on the browning of chips.

If the pigments responsible for the color are formed as the result of chemical reactions that take place during frying, the rate and extent of their formation would depend on several factors. Most important of these are the relative amounts and the type of the reactants found in different potatoes. The potato variety selected for chipping, if differing in these constituents, would determine the magnitude of such factors.

The investigation reported herein was designed to study the influence of the various types of sugars and free amino acids present in the potato tubers and their relation to the browning of potato chips.

EXPERIMENTAL

Four varieties were included in this study, namely: Katahdin, Russet Rural, Red Kote and Red Pontiac. The potatoes were furnished by Dr. J. Bushnell at the Ohio Agricultural Experiment Station, Wooster, Ohio. The potatoes grew under conditions typical of those usually prevailing in this part of the state. Potatoes were harvested as they reached full maturity. Two replicate samples, each consisting of 50 pounds, were taken at random from each plot and shipped to the Horticulture Department, The Ohio State University, where they were stored at room temperature (70° F.) for approximately 20 days. At the end of this period the potatoes of all varieties were divided into 3 lots. One lot was fried[c] and analyzed immediately and the other 2 lots were stored at 40° F. for 4 weeks. At the end of this period the 2 lots were removed from cold storage. One lot was immediately chipped and analyzed and the other stored at 75° F. for 4 weeks to be reconditioned. In each case 2 duplicates from every replicate were taken for frying and analysis. Each duplicate consisted of 10 pounds taken at random.

The potatoes were peeled in a Raynolds rotatory-type abrasive vegetable peeler. Peeled potatoes were halved longitudinally and sliced in a rotary type, hand-operated slicer. The resulting potato slices were thoroughly agitated in a pan of cold water to remove loose starch, removed to another pan of cold water, agitated again for a couple of minutes, whereafter they were spread on a dry, clean towel for 3 minutes to drain away excess moisture. They were then fried in fat (Primex) which had been previously heated to 375° F. At this point the heat was turned off, and the slices were fried to an endpoint determined by the cessation of bubbling in the frying medium.

Previous work by Habib and Brown *(7)* had indicated a highly significant correlation between the "L" Hunter readings of potato chips and the optical density of the pigments extracted from these chips when measured spectrophotometrically. These results indicated the validity of the Hunter "L" readings in predicting the relative concentrations of the coloring pigments resulting from frying. Thus, in this study, the colors of the chips were measured by the Hunter Color and Color Difference Meter.[d] The circuits of the color difference meter were initially balanced with an especially prepared ivory ceramic tile. The

[a] Journal Article No. 54-56, Ohio Agricultural Experiment Station, Project: Hatch 122-3.

[b] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, June 13, 1956, St. Louis, Missouri.

[c] Primex from the Procter & Gamble Company.

[d] Manufactured by Gardener Laboratories, Bethesda, Md.

circuit L, ^aL, and ^bL was used in balancing the instrument with the standard tile. Subsequently color specifications of the experimental samples were read directly and recorded in terms of L, ^aL, and ^bL. The values for this standard color for the L color scales are as follows:

$$L = +73.4 \qquad {}^aL = +1.00 \qquad {}^bL = +31.7$$

Potato tubers of the experimental samples were extracted with 80% ethanol and the resulting extracts were further used for the qualitative identification as well as the quantitative determination of sugars and amino acids by means of paper chromatography technique.

A 100 ml. aliquot of the alcoholic extract was concentrated at 50° C. to 20 ml. Ten milliliters of distilled water were then added to the residue and evaporation was repeated to 5 ml. The final residue was then made up to 10 ml. with distilled water. This solution was used for chromatographic analysis of both sugars and amino acids.

For the qualitative detection of the free amino acids, two dimensional, ascending paper chromatography was utilized, following the general technique of Williams and Kirby *(23)*, and employing the developers described by Cansden, Gordon, and Martin *(3)* as modified by Dent *(5)*. For the qualitative detection of sugars, descending paper chromatography was applied. The technique used was essentially that described by Hirst and Jones *(8)* as modified by Wolfrom and co-workers *(25)*.

For the quantitative determination of sugars and amino acids, the "area of spot" technique was employed, following the method described by Block *(1)*. At the completion of the chromatogram, the circumferences of the spots were carefully marked in pencil, and the areas were determined with planimeter. Four replicate analyses were carried out at each level of the standard and the experimental samples. It was necessary to establish a standard curve using known compounds which were L-arginine in the case of amino acids and D-glucose in the case of sugars. Six different concentrations of each, using the same volume each time were applied, and the chromatograms were developed exactly as the experimental samples. When the numerical values of areas developed from the standards were plotted on semilogarithmic paper against the concentrations, a straight line relationship was obtained. The standard curves were then used to read the concentration of the various sugars and amino acids of the experimental samples.

RESULTS

I. Free Amino Acids

Results of the two-dimensional chromatograms are presented in Tables 1 and 2, for the years 1954 and 1955, respectively. It should be mentioned here that in 1954 no attempt was made to analyze the tubers of all varieties after cold storage or after conditioning. Thus the data presented in Table 1 are those for the amino acids contents of the different varieties before cold storage only. There was some difficulty in interpreting the chromatograms in the aspartic acid-glycine region because of the distortion caused by the presenec of inorganic salts. Overlapping of asparagine with glycine was considerable, but the orange-yellow color of the asparagine spot was useful in distinguishing between these two spots. The chromatographic tests indicate that although all the varieties differed considerably in their content of free amino acids both quantitatively and qualitatively, the amino acids arginine, asparagine or aspartic acid, glutamine, valine, proline, and tyrosine appear to be present in all varieties. Tyrosine was not found in Russet Rural in 1954.

The absence of asparagine from the extract of some varieties is due most probably to the hydrolysis of this amide form to the corresponding aspartic acid during the preparation of the extract. Data in Table 2 show the results obtained from the varieties in 1955. Although the differences were considerable quantitatively between the two years' results, the differences qualitatively were very much less. Thus, the extracts prepared from the Katahdin variety that grew in 1954 contained the amino acids alanine, cystine and methionine—which were absent in the extracts prepared from the same variety in 1955. The latter (Katahdin, 1955) contained aspartic acid that was absent in the 1954 extracts. Likewise, Red Kote extracts of 1954 contained alanine and cystine that were absent in the extracts of 1955. Red Pontiac variety that grew in 1954 contained the amino acids leucine and methionine which were absent in the 1955 crop. The latter contained asparagine and glycine that were absent in 1954 crop. As for the Russet Rural variety the amino acids alanine, cystine and methionine were present in the 1954 crop and absent in the 1955 crop, but the amino acids asparagine and glycine were present in 1955 and absent in 1954. Two of the three basic amino acids that were present in 1954

TABLE 1

Amino acids contents of potatoes grown in 1954 as determined quantitatively by paper chromatography (mg. per 100 g. raw potatoes). Ave. 8 tests

Amino acids	Katahdin	Red Kote	Red Pontiac	Russet Rural
Alanine	20.3	30.1	0.0	22.3
Arginine	25.2	10.1	26.4	11.5
Asparagine	31.2	21.4	0.0	0.0
Aspartic acid	0.0	25.5	20.4	16.1
Cystine	20.3	20.4	0.0	13.5
Glutamine	31.2	22.4	40.6	28.8
Glycine	30.5	20.4	0.0	0.0
Histidine	14.1	0.0	20.4	0.0
Leucine	0.0	0.0	16.6	0.0
Lysine	0.0	20.6	20.4	13.5
Methionine	20.4	0.0	17.6	19.6
Proline	20.5	20.4	35.2	12.3
Valine	21.6	30.2	17.1	9.4
Tyrosine	8.2	10.3	9.6	0.0
Total	243.5	231.8	223.7	147.0

TABLE 2

Amino acids contents of potatoes grown in 1955 as determined quantitatively by paper chromatography (mg. per 100 grams raw potatoes). Ave. 8 tests

Varieties		Arginine	Asparagine	Aspartic Acid	Glutamine	Glutamic	Glycine	Histidine	Lysine	Proline	Tyrosine	Valine	Total
Katahdin	Before storage	27.5	16.5	9.0	18.5	12.2	7.5	18.5	0.0	18.5	7.5	9.0	144.7
	After storage	27.5	16.5	10.0	9.0	9.0	7.5	16.5	0.0	14.5	6.0	10.0	126.5
	After conditioning	0.0	9.0	9.0	18.5	12.2	0.0	0.0	0.0	27.5	0.0	0.0	76.2
Red Kote	Before storage	9.5	16.2	9.0	20.5	10.5	7.0	0.0	7.0	13.0	2.0	11.7	108.4
	After storage	11.5	13.5	10.2	10.7	8.5	10.5	0.0	0.0	13.0	2.0	9.7	89.6
	After conditioning	0.0	9.7	8.5	14.5	11.0	0.0	0.0	0.0	37.2	0.0	0.0	80.9
Red Pontiac	Before storage	27.5	23.0	7.7	18.0	7.0	6.0	22.5	13.0	14.0	7.0	10.7	156.4
	After storage	18.5	11.5	10.0	11.5	8.0	9.7	19.5	12.5	15.5	7.2	11.0	134.9
	After conditioning	0.0	12.0	7.7	16.5	7.7	6.2	0.0	0.0	12.0	9.2	18.0	89.3
Russet Rural	Before storage	16.5	22.5	10.0	18.5	12.2	12.2	0.0	0.0	18.5	9.0	16.5	135.9
	After storage	9.0	30.0	10.0	12.2	9.0	9.0	0.0	0.0	16.5	6.0	10.0	111.7
	After conditioning	0.0	9.0	9.0	18.5	12.2	0.0	0.0	0.0	27.5	0.0	0.0	76.2

were also present in 1955, and their ratios to total amino acids were comparable. These ratios can be calculated from data in Tables 1 and 2. Furthermore, it was found that cold storage at 40° F. except for lysine in Red Kote causes little changes in the amino acids contents both qualitatively and quantitatively. Conditioning at 75° F. however, resulted in the absence of many amino acids, particularly the basic ones as arginine, lysine and histidine along with a marked decrease in the contents of total amino acids.

II. Sugars

Data showing the separation of sugars and their quantitative determination are presented in Tables 3 and 4 for the years 1954 and 1955, respectively. It was necessary to use ultra-violet rays to locate and measure the spots of sucrose and maltose since they do not produce an easily detectable color with the color reagent used (trichloroacetic acid and aniline). There was some overlapping between glucose and xylose on some chromatograms, but the difference in the color of the spots (xylose is a red spot; glucose, a yellowish brown spot) helped in detecting both sugars.

TABLE 3

Sugar contents of potatoes grown in 1954 as determined quantitatively by paper chromatography (mg. per 100 g. raw potatoes). Ave. 8 tests

Sugars	Katahdin	Red Kote	Red Pontiac	Russet Rural
Maltose	12.0	0.0	51.0	0.0
Sucrose	47.0	66.0	66.0	28.0
Fructose	42.5	70.0	35.5	40.5
Mannose	35.0	0.0	33.5	0.0
Glucose	47.5	45.5	75.0	26.7
Xylose	24.5	42.8	60.0	0.0
Total	208.5	224.3	321.0	95.2

All varieties showed differences qualitatively and quantitatively in their contents of total and reducing sugars. Sucrose, fructose, and glucose were present in all 4 varieties. In addition xylose, mannose, and maltose were present in Katahdin and Red Pontiac. Red Kote contained xylose but not mannose or maltose. Data in Table 4 show the results obtained in 1955. Cold storage at 40° F. resulted in an increase in glucose and maltose and consequently in the total sugar contents; however, reconditioning resulted in the complete disappearance of maltose and a marked decrease in the rest of the reducing sugars. In all cases, however, sugar contents after conditioning were considerably lower than the corresponding contents before storage.

Color determination. Data in Table 5 show the color of the chips presented as Hunter "L" readings. In 1954 Russet Rural and Red Kote varieties produced lighter colored chips than either Katahdin or Red Pontiac. Red Kote and Red Pontiac varieties contained comparable amounts of sugar, and the difference in sugar contents was not of such a magnitude as to account for the difference in color. See Tables 3 and 4.) However, these two varieties differed considerably as to their content of basic amino acids. (See Tables 1 and 2.) The data secured in 1955 indicated again that the three varieties—Katahdin, Russet Rural, and Red Kote—produced chips lighter in color while the Red Pontiac variety failed to do so when potatoes were fried before storage. Also the comparison between the chips produced from Red Kote and Red Pontiac varieties in 1955 though not in 1954 indicate that Red Pontiac with higher amino acid content produced darker chips although the two varieties had comparable sugar contents. Furthermore, when potatoes were fried immediately after cold storage, the accumulation of reducing sugars, particularly glucose and maltose, caused the dark browning of the chips produced from all varieties. It seemed that the role of sugars in this particular situation was more predominant than the role of amino acids; i.e., there were no differences in color corresponding to the differences in amino acids. However, when potatoes were fried after conditioning, at which time both reducing sugars and amino acids had decreased considerably, then the color index of the chips increased correspondingly.

TABLE 5

Color of the chips produced from potatoes grown in 1954 and 1955 presented as Hunter "L" readings

Treatments	Varieties		Red Pontiac	Russet Rural
	Katahdin	Red Kote		
Crop of 1954 before storage	47.7	52.7	44.2	51.0
Crop of 1955 before storage	61.3	62.0	42.9	66.1
Crop of 1955 after storage	31.1	25.1	21.5	32.6
Crop of 1955 after conditioning	42.9	51.0	36.0	55.5

RESULTS AND DISCUSSION

The color of the chips presented as Hunter "L" readings was correlated with the total sugars, reducing sugars, and non-reducing sugars to give the following correlation coefficients in 1954: —0.02, —0.57 and +0.83 respectively. These figures indicate that total sugars could not be used as an index to predict the color of the chips. Although the correlation coefficient between reducing sugars and color is below the minimum 0.8 sometimes used for prediction purposes, it indicates that reducing sugars are of importance in the over-all reaction leading to color formation. The reducing sugars that were present in the varieties studied were maltose, fructose, glucose, mannose, and xylose. Of these reducing sugars, glucose and fructose were present in all varieties but in varying amounts. Mannose and the pentose sugar xylose were present in Katahdin and Red Pontiac. High amounts of xylose in Red Pontiac were associated with dark color chips; however, storage was found to cause slight changes in mannose, xylose, and fructose but resulted in a considerable increase in glucose and maltose (Katahdin excepted) presumably because these two sugars are among the com-

TABLE 4

Sugar contents of potatoes grown in 1955 as determined quantitatively by paper chromatography (mg. per 100 g. raw potatoes). Ave. 8 tests

Varieties		Maltose	Sucrose	Fructose	Mannose	Glucose	Xylose	Total
Katahdin	Before storage	24.0	90.0	49.0	36.7	43.2	39.0	281.9
	After storage	0.0	82.5	42.0	49.5	105.0	34.5	313.5
	After conditioning	0.0	44.2	32.2	0.0	24.0	0.0	100.4
Red Kote	Before storage	27.0	82.5	82.5	0.0	39.5	44.2	275.7
	After storage	30.0	90.0	64.5	0.0	72.0	34.5	291.0
	After conditioning	0.0	64.5	44.2	0.0	34.5	18.7	161.9
Red Pontiac	Before storage	49.5	60.0	67.5	44.2	49.5	82.0	352.7
	After storage	72.0	72.0	55.5	43.5	105.0	67.5	415.5
	After conditioning	0.0	49.5	44.2	0.0	52.5	27.0	173.2
Russet Rural	Before storage	44.2	39.1	36.7	0.0	36.7	0.0	156.7
	After storage	55.5	30.0	44.2	0.0	55.5	0.0	185.2
	After conditioning	0.0	27.0	24.0	0.0	28.5	0.0	79.5

TABLE 6
Correlation of different characteristics of raw potatoes vs. the color of the chips

Characteristics correlated	1954 Data		1955 Data	
	Correlation coefficient "r" values	Coefficient of determination (% values)	Correlation coefficient	Coefficient of determination (% values)
Hunter "L" readings vs. total sugars of raw potatoes	−0.20	4.0	−0.40*	16.0
Hunter "L" readings vs. reducing sugars of raw potatoes	−0.57*	32.5	−0.79*	62.4
Hunter "L" readings vs. non-reducing sugars of raw potatoes	+0.83*	68.8	+0.77*	59.3
Hunter "L" readings vs. total free amino acids of raw potatoes	−0.86*	73.9	−0.73*	53.3
Hunter "L" readings vs. basic free amino acids of raw potatoes	−0.95*	90.2	−0.80*	64.0
Hunter "L" readings vs. non-basic (neutral and acidic) amino acids of raw potatoes	−0.50*	25.0	−0.75*	56.3

* Designates significance at the 1% level.

pounds involved in the sugar-starch equilibrium. The results also indicate that the higher the content of non-reducing sugars, which was found to be sucrose in potato tubers, the higher the Hunter readings, i.e., the better the color of the chips ($r = +0.83$). This indicates that sucrose is not essential in the browning of the chips and furthermore, conditions under which chips are fried do not involve hydrolysis. The content of this sugar, however, is not markedly changed by cold storage but decreases in amount during conditioning. Results obtained in 1955 agree in general with those discussed above.

Upon correlating total amino acids content of the tubers vs. color of the chips produced therefrom in 1954, a correlation coefficient of —0.86 was obtained. This value is well above the 0.8 minimum value used for prediction purposes. This finding indicates the importance of amino acids in the reactions leading to color formation in the chips. Further evidence for the importance of amino acids in the browning of potato chips is present in the varietal difference especially between Red Kote and Red Pontiac in 1955 (See Tables 1 and 2), Red Kote containing less amino acids content and hence producing lighter colored chips than Red Pontiac. A higher correlation coefficient was obtained when basic amino acids contents were correlated with color. In this instance the value of the coefficient was —0.95. This would indicate that basic amino acids are more effective in color formation than neutral or acidic amino acids. The basic amino acids that were present in the varieties studied were histidine, lysine, and arginine. These amino acids were present in varying quantities. Cold storage slightly affected the amounts of these amino acids while conditioning at 75° F. resulted in their complete disappearance along with some of the other amino acids. Chips produced from all varieties were extracted with 80% alcohol and the extract was then condensed and analyzed by means of paper chromatography in the same manner used for raw potatoes. The results obtained indicated the disappearance of most amino acids except proline, glutamine, asparagine and aspartic acid. As would be expected the amino acids had been utilized in color formation. This might also indicate that the role of amino acids in the browning of the chips is more than that of catalyzer in the "caramelization" of sugars. Analysis of the 1955 data showed the same relationships with respect to amino acids contents.

A multiple regress analysis *(15)* was run to show the influence of reducing sugars, non-reducing sugars, basic amino acids, and non-basic amino acids (neutral and acidic amino acids) on color formation. The following regressing equation was obtained:

$$Y = 43.19 - 15.4X_a + 52.28X_b - 125.94X_c - 75.33X_d$$

where:

Y = Color presented as Hunter "L" readings.
X_a = Percent reducing sugar content of raw potatoes.
X_b = Percent non-reducing sugar content of raw potatoes.
X_c = Percent basic amino acids content of raw potatoes.
X_d = Percent "non-basic" (acidic and neutral) amino acids content of raw potatoes.

The results of the multiple analysis substantiate the results obtained by simple correlation between the color of the chips and the studied characteristics of raw potatoes. Thus, the higher the reducing sugars, the darker the color of the chips while the higher the non-reducing sugar content, the lighter the color of the chips. Also, the higher the content of amino acids, particularly basic amino acids, the darker the color of the chips.

SUMMARY AND CONCLUSIONS

In 1954 and 1955 experiments were designed to study the relation between the various types of sugars and free amino acids present in potato tubers and the browning of the chips. The sugars and amino acids were separated and their amounts determined quantitatively by paper chromatography.

The varieties studied showed differences in their contents of sugars quantitatively and qualitatively. The sugars sucrose, fructose, and glucose were present in all varieties, while xylose, mannose, and maltose occurred in some varieties only and in varying amounts. Cold storage at 40° F. resulted in an increase in glucose and maltose, but reconditioning at 75° F. resulted in the complete disappearance of maltose and a considerable decrease in other sugars especially the reducing sugars xylose and mannose.

Likewise, amino acids differed quantitatively and qualitatively from one variety to the other. Cold storage at 40° F. resulted in slight changes in amino acids but reconditioning at 75° F. resulted in disappearance of various amino acids especially the basic amino acids, lysine, histidine and arginine. The results indicated that, in general, low basic amino acids and low reducing sugars particularly pentose sugars in potato tubers are associated with light colored potato chips.

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Nutritive Value of the Proteins of Stored Instant Nonfat Dry Milk

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Storage deterioration of the protein nutritive value of nonfat dry milk is largely dependent upon the moisture content of the powder and the storage temperature. Henry and Kon *(2)* reported that the biological value of the proteins of dry skim milk decreased on prolonged storage. Hodson and Krueger *(7)* found that samples that browned during storage lost arginine, histidine, lysine, methionine and tryptophan. In contrast, samples with low moisture content stored in tin cans without access to atmospheric moisture and which did not brown suffered no significant losses of essential amino acids. Henry and Kon *(3)* confirmed the earlier work by showing with both rat nitrogen balance and rat growth methods that high moisture (7.3%) milk powder proteins lost biological value during storage for two years, whereas low moisture (2.9%) milk powder proteins did not. Microbiological tests by Henry *et al. (4)* indicated a loss of 40% of the lysine in high moisture powders.

Hare *(1)* showed that after 18 months' storage at 37° C. and under conditions of high humidity only 45% of the lysine of nonfat dry milk was recovered after acid hydrolysis of the protein and only 28% after enzyme hydrolysis. White *et al. (9)* showed that irrespective of the preheat treatments studied, dry whole milk of 1.9% moisture content could be stored for at least 6 months

at 37° C. in tin cans without loss of biological value or true digestibility of the proteins.

Although these reports clearly demonstrate the part played by moisture and/or storage conditions, specific data are still lacking on the type of nonfat dry milk offered at present in consumer-size packages in the United States. The older powders were often given high preheat treatments to render them especially suitable for bread and other bakery products. Newer powders are given low heat treatments to minimize cooked flavor and are "instantized" to afford ready mixing with water. They are usually packed in glass, laminated foil, or other containers quite impervious to moisture. In view of the wide-spread use of instant nonfat dry milk as a human food the collection of further data on the protein nutritive value of this product after storage seemed in order. Such a study was undertaken and will be reported here.

EXPERIMENTAL

Rat growth studies for protein nutritive value were conducted largely as reported earlier *(5, 6)*. Diets were fed at 9% protein level on a dry basis and were adjusted to the same fat and carbohydrate content. Fat addition was in the form of fresh butterfat. Six-week trials were run. In Trial I Sherman strain albino male rats from Rockland Farms were used. These rats were shipped in cold weather, and had to be delivered to the local express office by unheated truck since train express service has been terminated. Probably as a result of this unavoidable exposure, several cases of respiratory infection developed. The infection was first shown by almost complete refusal of food and by labored breathing. Examination of the lungs of the dead animals showed congestion. Most of the animals which recovered did not eat or gain normally. One rat in group 1 and two in group 3 died. Two rats in group 2 did not respond normally, and data from them were discarded. The remaining 8 or 9 animals in each group were in good health, and the data from them are considered reliable. The average starting weights for groups 1, 2, and 3 in Trial I were, respectively, 63.6, 63.7 and 63.2 g. Ten of the smaller rats were held on a cereal diet for a week then started as group 4 with an average weight of 66.0 g.

In Trial II weanling Long-Evans strain rats from our own colony were used. They were divided, as equally as possible according to litter, sex and weight into 3 groups of eight. The average starting weight of the rats in the 3 groups was 64.9, 64.5 and 65.0 g.

In Trial III the rats were similar to those in Trial II and were distributed into 2 groups of 10 rats each. The average starting weights were 69.8 and 69.3 g.

In Trial I a quantity of pasteurized (control) milk was frozen at the start and used throughout the experiment. This was used in the ration for group 1. Milk used in the ration for group 2 was a commercially produced instant nonfat dry milk (Commercial No. 1) which had been stored in glass jars for 9 months at room temperature. The instant nonfat dry milk sample used in the ration for group 3 was an experimental sample (Experimental No. 9) produced under semi-commercial conditions; it had been stored for 2 years at room temperature in glass jars with tight covers. This sample was known to have a protein nutritive value similar to pasteurized milk after instantizing but before storage *(6)*. Another experimental sample (Experiment No. 8) was used in the ration for group 4. This sample had been stored in a cardboard box with a mediocre foil overwrap for 2 years. The overwrap was a poor moisture barrier. The dry milk was badly caked, slightly browned, and quite unpalatable. Moisture content had increased from 3.4 to 5.7% during the first year of storage and was 5.8% at the start of the feeding trials. Moisture content of Commercial No. 1 was 3.3% and that of Experimental No. 9 was 3.7%.

For Trial II the ration for group 1 contained pasteurized milk. For most of the trial a frozen sample was used. For the remainder a fluid sample of pasteurized milk purchased locally was used. When the pasteurized milk was changed, it was necessary to make compensatory adjustments in the rations for groups 2 and 3, since it was desired to keep the rations as nearly comparable as possible. Group 2 ration contained Commercial No. 1 and group 3 ration Experimental No. 8 which are described above. In this case Commercial No. 1 had been stored about 11 months.

In trial III the ration for group 1 was fluid pasteurized milk purchased locally. Three different lots were used. Each sample was stored at 5° C. and used for a 2-week period and was then replaced with a fresh sample. The ration for group 2 contained a sample of instant nonfat dry milk (Experiment No. 1). This sample had been stored at room temperature for 2 years in a cardboard box with a good laminated foil overwrap. Moisture content increased from 3.1 to 4.3% during the first year and to 4.7% after 2 years but the sample remained white, free of lumps, and was reasonably palatable.

The rat growth method, which was used to study the protein quality of stored nonfat dry milk, is one of the most widely accepted methods of protein evaluation. It appears especially sensitive in detecting losses of protein nutritive value during the processing or storage of milk products.

The method is open to certain criticisms, however. One of these is the high requirement of the rat for the sulfur-containing amino acids. This requirement is not necessarily shared by other species. When the proteins of fresh milk are fed at low levels, it appears that the lack of sulfur-containing amino acids is the factor limiting rat growth. For this reason it seems desirable to supplement the rat growth data with some other means of protein evaluation. Microbiological assays for the amino acid, lysine, have been chosen for this purpose since information on loss of this amino acid seems of special value. The lysine of milk proteins seems particularly labile during milk processing or storage, and it is therefore a good general measure of amino acid loss. In addition milk proteins are especially rich in lysine and are thus suited for supplementing lysine deficient proteins such as those of cereals, so that knowledge of lysine content or losses is in itself important. Microbiological assays for lysine were made by the method of Stokes *et al. (8)*. One-gram samples were autoclaved in 10 ml. of 10% hydrochloric acid for 10 hours at 15 pounds pressure to hydrolyze the protein. In the assay the standard curve was plotted from 10 levels of lysine addition. Each level was run in quadruplicate. The samples were assayed at 5 levels. The pasteurized milk and the nonfat dry milk samples, Commercial No. 1 and Experimental 9, were assayed in quadruplicate at each level. Samples Experimental 1 and Experimental 8 were assayed in duplicate at each level. No values were discarded in calculating the averages.

RESULTS

Data from the 3 rat trials are summarized in Table 1.

In the first trial, data for the first 3 groups will be considered together. The difference in average gains in weight are not significant. F is equal to 0.19, and F equals 3.42 is required for significance at the 95% level. The differences in average protein efficiency expressed as grams gain per gram protein consumed are also not significant.

As mentioned above, the rats and treatment for group 4 were not entirely comparable to the other groups, since the former were selected because of their small size and were held a week before starting on experiment. However, the rats in group 4 responded unusually well and had the best average weight gain and protein efficiency at the end of 6 weeks. For lack of a better control group the analysis of variance comparison was made to group 1. For the average weight gains F is equal to 1.236 and for the protein efficiency, 0.429. F equals 4.55 is required for significance at the

TABLE 1
Protein efficiency data on stored nonfat dry milk

Trial	Group number	Sample identification	Storage period	Final moisture in percent	Average gain in rat weight in grams	F	P. E.-grams gain per gram protein consumed	F
I	1	Pasteurized milk	.....	....	83.9		2.34	
	2	Comm. No. 1 NFDM	9 months	3.3	78.9		2.17	
	3	Exp. No. 9 NFDM	2 years	3.7	83.5	0.19[1]	2.28	0.51[1]
	4	Exp. No. 5 NFDM	2 years	5.8	93.8	1.24[2]	2.41	0.43[2]
II	1	Pasteurized milk	.....	....	82.5		2.42	
	2	Comm. No. 1 NFIM	11 months	3.3	97.2		2.62	
	3	Exp. No. 8 NFDM	2 years	5.8	88.9	1.63	2.46	0.81
				....				
III	1	Pasteurized	.....		76.3		2.55	
	2	Exp. No. 1 NFDM	2 years	4.7	81.1	0.83	2.46	0.81

[1] Comparison of groups 1-3.
[2] Comparison of groups 1 and 4 only.

95% level. Even though the data of groups 1 and 4 are not strictly comparable, there is little to suggest that the protein nutritive value of nonfat dry milk No. 8 has been diminished by the gain in moisture, lumping, or slight browning which occurred during storage.

Trial II was an effort to verify and strengthen the data of Trial I. Again the results indicated that no damage was done to the protein nutritive value under the storage conditions studied. During the 6 week's trial the rats in group 1 (pasteurized milk) gained an average of 82.5 g., those in group 2 (Commercial No. 1) gained 97.2 g., and those in group 3 (Experimental No. 8) gained 88.9 g. Although these differences are large, the variance within groups is also large and the differences are not statistically significant. F equals 1.63, and F equals 3.47 is required for significance. The differences in protein efficiency are somewhat more uniform and are also not significant. F equals 0.81, and F equals 3.47 is required for significance. A separate comparison was made between groups 1 and 2. This difference is also not significant. F equals 1.39, and F equals 4.32 is required for significance. The fact that in the two trials the difference between groups do not fall in the same order further suggests that the differences are not significant.

Like the data of the first two trials, data of Trial III also indicate that no damage was done to the protein nutritive value by storage under the conditions studied. Rats fed the pasteurized milk protein gained an average of 76.3 g. while those fed nonfat dry milk protein gained 81.1 g. An analysis of variance indicates the difference is not significant. F is equal to 0.83, and F equals 4.41 is required for significance at the 95% level. The protein efficiencies are in the reverse order, i.e., 2.55 for the pasteurized milk group and 2.46 for the nonfat dry milk group. This difference is not significant. F is equal to 0.81, and F equals 4.41 is required for significance.

Lysine data are summarized in Table 2. Lysine content of the proteins of the pasteurized milk and of the 4 nonfat dry milks are all quite similar. Results of the microbiological assay were very uniform in this case, and an analysis of variance indicates that the small differences cannot be entirely assigned to the variations in the assay, and hence must result from storage treatment or from minor uncontrolled variations. F equals 3.50. F equals 2.50 is required for significance at the 95% level and F equals 3.60 is required for significance at the 99% level. The proteins of Commercial Sample 1 have a higher lysine content than the proteins of the pasteurized milk. This difference is not significant. F equals 1.70. F equals 3.98 is required for significance at the 95% level. Lysine content of the proteins of Experimental Sample 9 is almost identical with that of the proteins of pasteurized milk. Thus, the samples

TABLE 2
Comparison of lysine content of pasteurized milk and stored instant nonfat dry milk

Type of milk	Sample number	Approximate storage period	Final percent moisture	Percent lysine in protein
Fluid pasteurized	....	A few days	....	7.77
Nonfat dry milk	Comm. No. 1	1 year	3.3	7.96
Nonfat dry milk	Exp. 9	2 years	3.7	7.78
Nonfat dry milk	Exp. 1	2 years	4.7	7.34
Nonfat dry milk	Exp. 8	2 years	5.8	7.53

with low moisture, retained their lysine during storage. There is some indication for a trend toward lysine loss in the samples which gained moisture. The lysine content of the proteins of Experimental Sample 1 is 5.5% lower than that of the proteins of pasteurized milk and this difference is significant. F equals 5.50, and F equals 3.98 is required for significance at the 95% level. However, the proteins of Experimental Sample 8 which appeared to have the greatest storage deterioration had a lysine content only 3.0% lower than the proteins of pasteurized milk, and this difference is not significant. F equals 1.73, and F equals 3.98 is required for significance at the 95% level. Thus, only a trend is indicated but this is in agreement with earlier data *(7)* which indicated a marked lysine loss when browning was severe. In no case do the present data indicate a serious lysine loss. It should be pointed out that certain differences exist between the powders used in the present study and those used 10 years ago *(7)*. The powders discussed here were low heat, instantized products prepared in stainless steel equipment from raw milk of high quality. Those used 10 years ago were high heat powders, not instantized, and were prepared in plants that probably still possessed some copper processing equipment. However, the average lysine contents of samples of the present study and those of the "fresh" samples studied earlier *(7)* are quite similar i.e., respectively, 7.68 and 7.83% of the proteins.

SUMMARY

The protein efficiencies for rat growth of pasteurized milk proteins and those of instant nonfat dry milks stored under favorable conditions for one or two years are similar.

The lysine contents of the proteins of pasteurized milk and of properly stored instant nonfat dry milks are similar.

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Microorganisms of Significance in Frozen Citrus Products[a]

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It is unfortunate, considering the conscientious, vigorous effort of the food industry to produce frozen citrus products of uniform, high quality *(9, 36, 37)*, comparable to the fresh form of one of the "basic 7" foods in the human diet *(15, 40)*, that confusion has arisen concerning the significance of various groups of microorganisms in these products. Since frozen citrus concentrates are acid foods *(11, 34, 35)*, are protected from contamination by being hermetically sealed in cans, and are produced with careful attention to the effect of each step in the process on microbial content *(46)*, efforts to establish microbiological standards for these products should begin with a thorough awareness of these factors *(2, 3, 12, 13, 19, 52)*. The purpose of this paper is to record the essential criteria regarding the significance of such organisms.

Significance of microorganisms in frozen citrus products. Early in the development of the frozen citrus concentrates industry, much time and effort was given to a study of the coliform bacteria in frozen orange concentrate. Then, during the 1949-50 season, it was discovered that the lactic acid bacteria caused acute spoilage of orange juice in process of concentration *(19, 20)*. At nearly the same time, nonmicrobial enzymatic gelation of orange concentrate and separation of the pulp and "cloud" in reconstituted juice became a serious problem *(1, 38, 43)*. Finally flash heating was adopted to control gelation and solids separation. This manipulation caused a marked reduction but did not eliminate viable microorganisms from the finished product *(29, 46)*. Unheated fresh juice still had to be added to concentrate just prior to canning to restore "fresh" flavor and aroma constituents lost during vacuum concentration. Therefore, the microbial spoilage problem still remained, and the question of the significance of the various groups of microorganisms found in or on citrus had to be resolved. What place did the traditional microbial indices of fecal pollution [the coliform bacteria, the fecal streptococci, and *Clostridium perfringens (C. welchii)*] play in sanitary control in the frozen citrus products industry? Should the industry continue to use the traditional indices of sanitation and minimize other bacteria known to cause acute spoilage of their product? One possible solution is indicated by review of the facts accumulated until now.

Significance of coliform bacteria. Coliform bacteria and related types (e.g. *Erwinia*) are frequently present on or in oranges even before they are harvested. Furthermore, laboratory tests have demonstrated that coliform organisms sometimes are present in orange juice in spite of rigorous aseptic care in harvest and regardless of the amount of antiseptic washing given the fruit before extraction *(5, 12, 42)*.

The rather low incidence of coliform bacteria in orange concentrate tested routinely in a number of plants in Florida is shown by the data in Table 1. The data of Wolford *(48, 49)* substantiates these observations. Analysis of these studies shows that a very high percentage of false positive reactions occur in Florida

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(Table 1) and in California *(48, 49)*. Martinez and Appleman *(27)* also have encountered false positive coliform reactions caused by yeasts.

The unreliability of the "standard" coliform test as applied to citrus has been shown by Wolford *(50, 51)* to result from the natural fermentable constituents of the orange juice transferred to the test medium with the inoculum. To circumvent this inconsistency an attempt was made to use the boric acid lactose broth medium *(47)* for detection of *Escherichia coli*, rather than total coliform bacteria. A higher percentage of positive presumptive tests for *E. coli* could be confirmed but trouble was experienced with *Aerobacter* types. This was found to be due, in part at least, to the fact that *Aerobacter* types could be trained to resist 0.325 percent boric acid and also to grow at 42.5-43.5° C. (108.5-110.3° F.) (unpublished data of Vaughn and Krumperman). Levine *(50)* suggested that the presence of naturally occurring citrates could favor the more frequent recovery of *Aerobacter* from the positive presumptive tubes of boric acid lactose broth than was reported by Vaughn, Levine and Smith *(47)* for water.

The high percentage of anomalous results obtained in frozen citrus products suggests the unreliability of the coliform tests as used under "standard" conditions. Furthermore, there has been no observable significant correlation between the presence of coliform bacteria in the product and sanitary surveys made of the citrus plants that produced the product tested *(8, 32)*. In view of the chemical nature of the product *(6, 11, 35, 50)* it becomes questionable whether any significance can be attached to the presumptive coliform test for frozen orange concentrate.

There are no data to indicate that the coliform bacteria actually can grow in citrus juices. On the other hand there is considerable evidence to show that coliform bacteria can retain their viability for extended periods in frozen orange concentrate but die off rapidly in fresh or reconstituted juices *(12, 17, 45)*. Under these conditions it should be apparent that the coliform index of frozen concentrate would be of no value for detecting possible fecal contamination that might contain pathogenic enteric (intestinal) bacteria *(Salmonella* and *Shigella)*, especially since the product is almost always stored more than a week before introduction into commercial channels *(17)*. Neither *Salmonella* nor *Shigella* types, unlike species of *Aerobacter* and *Escherichia* can survive for extended periods in the acid environment of either citrus juices or concentrates *(2, 17)*. Furthermore, the spores of *C. parabotulinum*, types A or B, apparently cannot germinate in this acid environment even though they may be present *(41)*.

Significance of streptococci. Hunter *(23)* declared that "determination of the presence of coliform organisms does serve a very useful purpose when there has been sufficient study of the methods of production and handling of the class of food product under control to lay the basis for interpretation of the significance of members of the coliform group as a whole or of genera or species within the group. Until this basis is laid, the very ubiquity of the group places a handicap upon the interpretation of results obtained by laboratory examination." The statement of Hunter, paraphrased to substitute "fecal streptococci" for "coliform organisms" is equally applicable to the determination of the significance of streptococci in frozen citrus products. The "very ubiquity" of the fecal streptococci suggests that they probably are not good indicators of sanitation. As a matter of fact, there is no evidence to indicate that fecal streptococci can grow in frozen citrus products *(14, 17, 18, 24, 25, 26)* even though they may be present and survive for a period of at least 147 days *(18)*. Furthermore, the rather small amount of work done in connection with their occurrence in frozen citrus products *(14, 17, 18, 24, 25, 26)* would not, in any case, be considered a sufficient study, according to the statement of Hunter *(23)*.

It is known that other organisms such as the lactobacilli and propionic acid bacteria can be and are rather easily isolated from the same sources as the fecal streptococci (i.e., feces, skin, and body cavities of humans). This fact poses the question of the normal ecology of either the fecal streptococci, lactobacilli, or propionic acid bacteria.

Furthermore, to quote Thatcher *(44)* "Practical considerations may demand some tolerance of enteric bacteria," a point that also seems to be implied by a statement made by Dack *(12)*. The latter authority urges public health authorities "to take cognizance of the fact that our environment and the non-sterile foods in our habitat contain enteric bacilli, regardless of our efforts to eliminate them."

According to Dack *(12)* "Millions of cans of frozen concentrated orange juice have been consumed without causing any known cases of enteric infections." Since there is no concrete evidence of a health hazard of microbial origin, we submit that microbiological significance of any given group of organisms in frozen citrus products should be influenced by the probability of that specific microbial group being able to survive and grow

TABLE 1

Incidence of coliform bacteria in frozen orange concentrate [1]
(Four typical Florida plants)

Plant	Packing seasons reported	Number of samples examined	Presumptive tests boric acid lactose broth		Confirmed tests		False positive tests	
			Number positive	Percent positive	Number positive	Percent positive	Number	Percent of presumptives
A	2	3,981	65	1.6	26	40	39	60
B	2	4,618	50	1.1	0	0	50	100
C	2	3,562	0	0	0	0	0	0
D	1	450	116	25.8	15	13	101	87
Totals		12,611	231	1.8	41	17.7	190	82.3

[1] Compiled from unpublished data furnished by members of Subcommittee on Microbiology, IFT Citrus Products Committee.

in the particular frozen citrus product. The organisms known to grow in citrus juices include yeasts, molds, acetic and lactic acid bacteria.

Significance of yeasts and molds. Citrus fruits may be invaded or contaminated by yeasts and molds even before harvest *(21, 32)*. To prevent entry of moldy, rotten or otherwise damaged fruit into the concentrating plant, careful sorting and elimination of all defective fruit is the last step before washing and sanitizing fruit. Routine microscopic examination of the extracted juice serves as a control in the efficiency of sorting. Yeasts and molds, unlike the lactic acid bacteria, usually do not grow rapidly enough to cause spoilage under the conditions that normally prevail during vacuum concentration of the juice.

Significance of the acetic acid bacteria. Organisms of the genus *Acetobacter* also grow well in single strength or partially concentrated orange juice, and produce sour, buttermilk to sauerkraut-like flavors and odors *(7)*. Representative strains also produce diacetyl from the major constituents of orange juice *(6)*. However, this group is not considered to be a useful index of product quality. The fastidious growth requirements of many strains are a major disadvantage for quality control. Their frequency in normal citrus is low as compared with the lactic acid bacteria. Also they are strict aerobes and therefore are not significant indices of product quality in the concentrator operated under high vacuum.

Significance of the lactic acid bacteria. Intermittent spoilage of orange concentrate characterized by the development of flavors and odors resembling those of buttermilk was first observed in Florida during the 1949-50 season. The undesirable "buttermilk" flavors and odors were found to result from undesirable increases in populations of lactic acid bacteria during preparation and concentration of the juice *(19, 20, 30)*. It is well established that various species of *Lactobacillus* and *Leuconostoc* are responsible for most of the outbreaks *(19, 20, 30)*. It has been established that diacetyl is responsible for part of the undesirable flavor produced by lactic acid bacteria *(6, 10, 22)*, and that diacetyl is produced from one or more of the major constituents of orange juice by these bacteria *(6)*.

The lactic acid bacteria may be present on and in the fruit at harvest. Therefore, it is very important that careful sorting and efficient washing of the citrus be conducted because these organisms grow well in single strength and partially concentrated orange juice. Care also must be taken to prevent a build-up in population of these bacteria at any point in the process. The important focal points that serve as sources for infection of the single strength or partially concentrated juice include the juice extractors, finishers, fresh juice holding tanks, evaporator condensers, and the cooling and regenerative sections of the plate type heat exchangers *(29)*. As a control measure to determine degree of contamination, plate and direct microscopic counts are made on the product at regular intervals during its concentration. These tests are frequently supplemented by qualitative and quantitative tests for diacetyl *(6, 10, 22)*.

Since enteric organisms as a group have not been shown to be important indicators of sanitation in citrus products and since there is no apparent health hazard of bacterial origin, the citrus industry has focused its attention on those organisms that survive and grow in citrus juices. Of these, the lactic acid bacteria as a group grow rapidly and cause acute spoilage, and therefore have the most significance from the standpoint of production of products of high quality.

DISCUSSION

The widespread tendency toward establishment of microbiological standards for foods *(28, 44)* must be tempered by consideration of the chemical and physical characteristics of different foods, else, through the results of unrealistic thinking, unattainable tolerances may be established.

Frozen citrus products are examples of non-sterile acid foods which do not present any known bacterial health hazards *(12)*, yet are faced with the pressures of such unrealistic thinking. If the proponents of the traditional indices of sanitation (i.e., coliform bacteria *vs.* enterococci *vs. C. perfringens*) were to undertake sanitary surveys of the citrus groves and plants in both California and Florida, they might realize the futility of their stand.

After six intensive years of study, both major geographical segments of the U. S. citrus industry have concluded that lactic acid bacteria are the best index of processing sanitation. If these organisms are controlled, high quality is maintained; if not, an inferior product is produced.

SUMMARY

A review of the significance of microorganisms in frozen citrus products has revealed no known health hazard of bacterial origin. Therefore, the traditional bacterial indices of sanitation are not applicable. Instead, it is concluded that lactic acid bacteria constitute the best possible index of processing sanitation now known for production of high quality frozen citrus products.

Acknowledgments

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Stability of Frozen Concentrated Orange Juice. III. The Effect of Heat Treatment In the Production of High-Brix Frozen Concentrate[a, b]

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PREVIOUS PAPERS IN THIS SERIES have dealt with the effects of different times and temperatures of heating on enzyme inactivation and cloud stability of frozen Pineapple and Valencia orange concentrates *(5)* and a comparison of a small tube heat exchanger, plate type pasteurizer, and a steam injection heater *(6)* for enzyme inactivation and improving cloud stability. In the current study a comparison was made of the cloud stability of 42° Brix and 58.5° Brix concentrates and the effects of heat treatment and methods of preparation on the cloud stability of 58.5° Brix concentrates.

Increasing interest in high Brix (58.5° Brix or five plus one) frozen concentrated orange juice has been shown during the last few years by processors and shippers. The Florida legislature has passed an amendment to the Florida Citrus Code *(4)* permitting experimental production of five plus one concentrated orange juice for institutional and similar use.

When early workers selected 42° Brix as the final concentration of frozen concentrated orange juice, the incorporation of a liberal amount of fresh cutback juice for flavor was a principal consideration and it was not anticipated that cloud loss or gelation would be serious problems. However, product deterioration resulting from abnormal temperatures during storage and distribution *(9)* has assumed economic significance. Concentrates of higher concentrations have greater cloud stability and less tendency to gel and therefore offer possibilities for industrial development.

Cotton, Roy, Brokaw, McDuff, and Schroeder *(2)* noted increasing cloud stability in orange concentrates with increasing concentration above 42° Brix.

Bissett, Veldhuis, and Rushing *(1)* investigated the heat treatment of concentrates of different strengths. The concentrates were heated, filled hot into cans, and cooled. Concentrates of 57° Brix were more stable than those of lower concentration. A treatment temperature of 160° F. was sufficient to provide cloud stability for 240 days at 35° F. for the 57° Brix concentrate while 190° F. was required to provide equal stability for the 42° Brix product.

Rice, Keller, McColloch, and Beavens *(8)* prepared 4-, 5-, 6-, and 7-fold orange concentrates by dilution with water or further concentration of commercial 6-fold concentrate and observed cloud stability in 20° and 40° F. storage. In all cases cloud stability increased with increased concentration.

DuBois and Murdock *(3)* observed not only that high-Brix products are inherently more stable than 42° Brix concentrates but also that in the preparation of high-Brix concentrate, heating of 18° Brix materials to 150° F. for 10 sec. provided stability for at least two weeks in 40° F. storage.

The studies reported here were designed to supplement the above information on PE (pectinesterase) inactivation and cloud stability in three respects: (1) a direct comparison of 42° Brix and 58.5° Brix concentrates prepared from both heated and unheated juces; (2) a study in heat treatment of a portion of the evaporator feed juice in the preparation of stable high-Brix concentrates; and (3) the effect of varying the degree of concentration in the evaporator in the preparation of high-Brix products. Samples were evaluated for stability during a six-week storage period at 40° F.

PROCEDURE

Since the experimental plan called for packs of such proportions that several days would be required to prepare the concentrates, it was necessary to use every means possible to reduce raw material variables. This was accomplished by having a suitable quantity of Valencia oranges picked from a block of trees selected for uniformity, delivered directly to the laboratory, and placed at once in 40° F. storage. Sufficient fruit for a day's operation was removed from storage in a random manner, washed, and the juice extracted in a Rotary Juice Press.[c] Rag and seeds were removed in a finisher equipped with an 0.033" screen. The screened juice was pumped directly to a coldwall tank and held at 40° F. until required for processing. The heat stabilization treatment was applied to single strength juice in either a small tube turbulent flow or a commercial plate type heat exchanger as described in a previous publication *(6)*.

The small tube unit required 0.8 sec. to raise the juice to the desired temperature while the plate type required 25 sec. In either case the juice was held at the designated temperature for 5 sec. and then quickly cooled. Treatment temperatures used in these experiments included 150°, 160°, 170°, and 180° F. A juice supply of 20 to 25 gallons for each treatment variable was sufficient to assure a two- to three-gallon recovery of concentrate from the evaporator.

For comparison of 42° and 58.5° Brix concentrates, heat treated juices and an unheated control were concentrated to 55° to 68° Brix at 75° F. in a falling film evaporator and then cut back to 42° and 58.5° Brix, respectively, with unheated juice, filled and closed in 202 x 214 plain cans and frozen under an air blast at 0° F.

In studies designed to determine the minimum heat treatment required to provide stability in high-Brix concentrates under standard conditions of this experiment, portions of 68° Brix concentrate were prepared from both heated and unheated juices. Portions of concentrate from each heat treatment were mixed with concentrate from unheated juice to give 68° Brix concen-

[a] A partial report on work conducted at the U. S. Citrus Products Station, Winter Haven, Florida, under a Memorandum of Understanding between the Continental Can Company, Inc., New York, N. Y., and the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.

[b] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 13, 1956.

[c] The mention of trade products does not imply endorsement over similar products not mentioned.

trate containing 100, 75, 66, 50, 33, 25, and 0% heat stabilized fractions. These were all cut back to 58.5° Brix with unheated juice, canned, and frozen.

The effect of concentrate to cutback ratio was studied by preparing 62°, 64°, and 66° Brix concentrates cutting back in each case to 58.5° Brix with unheated single strength juice, canning, and freezing. Products were thus prepared for each temperature of heat treatment in addition to unheated controls.

All samples were tested for PE activity by a modified method described by Bissett, Veldhuis, and Rushing *(1)*. Cloud stabilities were estimated by transferring samples of all final products from 0° to 40° F. storage, and testing at regular intervals up to 42 days. Cloud index was determined on reconstituted concentrate by the method of Loeffler *(7)* using an Evelyn colorimeter [4] equipped with a No. 720 filter. High cloud index (light transmission) readings indicate clarification or poor cloud stability. A visual method of evaluation of changes was also used in which a portion of the reconstituted 12° Brix juice was placed in a test tube and stored at 40° F. After 24 hours the sample was visually graded as being either stable, partially separated, or completely separated. By using both colorimeter and visual methods, it was possible to correlate the colorimeter values with visual cloud loss of the juice. Such comparisons made it possible to establish ranges of light transmission corresponding to the visual grades.

The 42° and 58.5° Brix concentrates were tested for flavor differences using a variation of the latin square design. A group of 16 to 20 trained persons who had been selected for their ability to detect small flavor differences was used in forming the taste panel. Samples were graded on an hedonic preference rating scale of 1 to 9. According to this method a numerical value of 5 would indicate neither like nor dislike of the product.

The average analysis of the Valencia orange juice used in these investigations was as follows: Brix, 11.7°; anhydrous citric acid, 0.81%, pH, 3.71; suspended solids, 10.0%; and oil, 0.050%.

RESULTS AND DISCUSSION

PE and cloud stability data for single strength juices and concentrates comparing 42° and 58.5° Brix concentrates are given in Table 1. For each pasteurization temperature the same juice was used to prepare both types of concentrate so the PE values of the heated juices are the same. Small variations between pairs of the 55° and 68° Brix concentrates are attributable to normal deviations in the method of enzyme assay. The PE values in the 42° Brix concentrates were higher than those in the corresponding 58.5° Brix concentrates because of the greater proportion of unheated cutback juice. Progressively higher heat treatments gave increasingly greater enzyme inactivation, but inactivation was not complete even at 180° F. The percentage activity remaining ranged from 13 to 50% of that in the original juice.

Stability in terms of weeks in 40° F. storage during which there was no visual evidence of cloud loss is also shown in Table 1. Unheated controls failed to retain their cloud stability during the first week. The 42° Brix heated products remained stable for periods of one to 3 weeks depending upon the treatment temperature used and all 58.5° Brix concentrates representing treatment temperatures of 150° to 180° F. remained completely stable for the entire 6-week observation period.

Cloud stability curves for the unheated and heated 42° and 58.5° Brix concentrates are presented in Figures 1 and 2. In comparing visual cloud stability data with percent light transmission data obtained with the colorimeter, it was noted that visual cloud loss occurred when the light transmission value rose to about 34% and complete cloud loss when the light transmission values were above about 60%. Intermediate values generally indicated partial separation. On the basis of this comparison each of the figures has been divided into 3 zones designated as stable, partially separated, and completely separated.

The 42° Brix unheated control showed signs of cloud instability soon after being placed in 40° F. test storage as shown in Figure 1. Progressively higher heat treatments resulted in increased cloud stability in the 42° Brix products. However, even a 180° F. treatment failed to stabilize the cloud for more than 3 weeks. The 58.5° Brix unheated control did not lose its cloud as

TABLE 1

A comparison of the effect of heat treatment in a plate type heat exchanger on PE inactivation and cloud stability in 42° and 58.5° Brix concentrate made from Valencia orange juice

Treatment temperature	Final Brix	PE in juice	PE in evaporator product	PE in final product	Activity remaining [1]	Weeks stable at 40° F. (visual)
° F.		*PEu./ml. x 10^4 per 12° Brix*			%	
Control	42°	23.5	16.2	17.9	....	<1
	58.5°	25.6	17.5	17.0	....	<1
150	42°	8.5	8.6	8.8	50	1
	58.5°	8.5	8.3	7.2	41	>6
160	42°	3.9	3.1	4.8	27	2
	58.5°	3.9	3.3	3.1	18	>6
170	42°	1.8	1.8	3.3	19	2
	58.5°	1.8	1.9	2.3	13	>6
180	42°	1.9	1.5	2.7	15	3
	58.5°	1.9	1.5	2.2	13	>6

[1] Percent activity remaining calculated from control average of 17.5 PEu./ml. x 10^4 per 12° Brix.

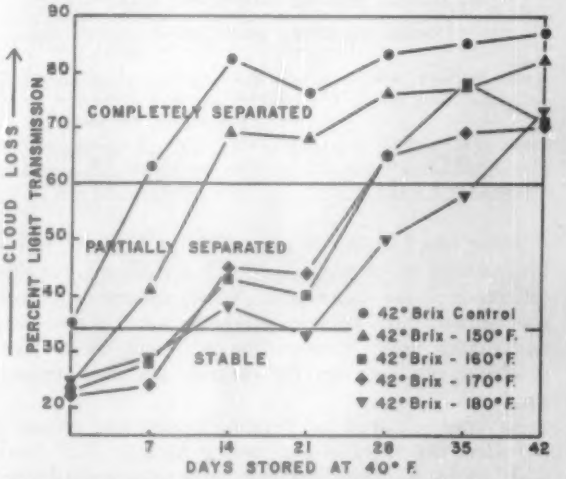

Figure 1. The effect of heat treatment on the cloud stability of 42° Brix concentrates.

[4] In order to permit a comparison of cloud values with other studies, the readings of standard bentonite suspensions obtained with the instrument used are given below (A Proposed Standard for Designation of "Cloud" in Citrus Juices by V. J. Senn, W. D. Murray, and R. T. O'Connor, ARS-72-8, Agricultural Research Service, U.S. Dept. of Agriculture, Oct. 1955. Processed):

Bentonite, g./l.	0.0	0.5	1.5	2.5	3.5	4.5
Percent transmittance	100.0	71.2	46.8	31.5	23.0	17.8

rapidly as did the 42° Brix control, as shown in Figure 2. The 150° F. heat treatment in the preparation of 58.5° Brix products resulted in virtually no cloud loss throughout the 42-day test period at 40° F. Heat treatments of 160°, 170°, and 180° F. also gave products of similar high cloud stability.

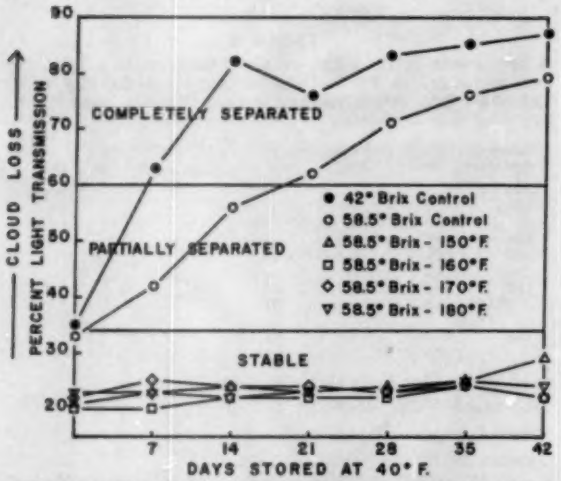

Figure 2. The effect of heat treatment on the cloud stability of 58.5° Brix concentrates.

Data for cloud stability and PE inactivation for the small tube heat exchanger were very similar to that obtained with the plate type heat exchanger and, for the sake of brevity, are not presented in detail. In each case the 58.5° Brix concentrates were more stable than the corresponding 42° Brix concentrates. Heat treatment of 150° F. gave a high-Brix concentrate of complete cloud stability throughout the 42-day test period at 40° F.

Results of flavor comparisons between 42° and 58.5° Brix concentrates are shown in Table 2.

TABLE 2

A comparison of flavor between 42° and 58.5° Brix concentrates receiving various heat treatments

		Control	150° F.	160° F.	170° F.	180° F.	Average
Small tube	42°	6.3	6.3	6.3	6.2	6.0	6.2
	58.5°	6.2	6.2	6.3	6.4	6.3	6.3
Plate type	42°	6.3	6.3	6.2	6.2	6.0	6.2
	58.5°	6.3	6.3	6.0	6.3	5.9	6.2
Average		6.3	6.3	6.2	6.3	6.1	

There was a slight tendency toward a lower score for concentrates which received 180° F. treatments, but this difference is not significant. No significant difference was demonstrated between products from the two heat exchangers used, between the 42° and 58.5° Brix products, nor between the controls and heat treated products.

Samples prepared by blending heated and unheated 68° Brix concentrates and cutting back to 58.5° Brix with unheated single strength juice as previously described were examined for PE activity and cloud stability. The data are presented in Table 3. As would be expected PE activity decreased with increased treatment temperature and increased as the proportion of heated concentrate decreased. When as little as 50% of the 68° Brix concentrate had been heated to 150° F. the product retained its cloud stability for the full 6 weeks of observation, even though it retained 74% of its original PE activity. Use of higher heat treatment temperatures produced concentrates of similar stability but demonstrated no particular advantage.

In Figure 3 stability curves are shown for 58.5° Brix products containing varying proportions of heated (150° F.) to unheated 68° Brix concentrates. The control containing no heated material remained stable for approximately 10 days; that containing 25% heated concentrate for 25 days; and that containing 50% heated concentrate continued to retain its cloud for the full 42-day observation in 40° F. storage. Although the curve indicates stability of the product containing 33% heated concentrate for the entire period of observation, visual examination showed that partial separation occurred in some samples at 4 weeks. There was no advantage in use of treatment temperatures above 150° F. in the preparation of high-Brix products and only one-half the evaporator feed juice needed to be so treated in order to produce a stable concentrate.

The PE value for the experiments in which the degree of concentration before cutting back varies in the range

TABLE 3

The effect of blending heated and unheated 68° Brix concentrates used in preparation of 58.5° Brix products

Percent of heated juice in 68° Brix conc.[1]	PE. in juice	PE. in evaporator product	PE. in final product	Activity remaining[2] %	Weeks stable at 40° F. (visual)
		PEu./ml. x 10^4 per 12° Brix			
		150° F.			
100	10.2	7.5	8.2	53	>6
75			8.1	52	>6
66			11.2	72	>6
50			11.4	74	>6
33			12.4	80	4
25			14.9	96	3
0	23.0		15.5	....	1
		160° F.			
100	3.7	3.2	3.1	20	>6
75			7.2	46	>6
66			10.7	69	>6
50			9.2	59	>6
33			9.0	58	3
25			11.0	71	3
0	23.0		15.5	....	1
		170° F.			
100	2.7	1.9	2.7	17	>6
75			4.8	31	>6
66			7.3	47	>6
50			9.8	63	>6
33			10.3	66	4
25			9.7	63	4
0	23.0		15.5	....	1
		180° F.			
100	1.9	1.5	2.0	13	>6
75			4.9	32	>6
66			6.1	39	>6
50			7.7	50	>6
33			10.4	67	4
25			11.9	77	3
0	23.0		15.5	....	1

[1] Heated and unheated concentrates (at 68° Brix) were used in various proportions. All combinations then cut back with single strength unheated juice to 58.5° Brix.

[2] Percent activity remaining calculated from control average of 15.5 PEu./ml. x 10^4 per 12° Brix.

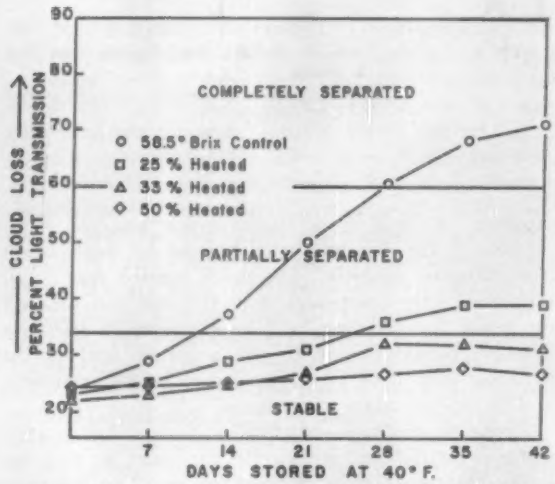

Figure 3. The effect of varying proportions of heat treated juice (150° F.) on the cloud stability of 58.5° Brix concentrates.

of 62° to 66° Brix are given in Table 4. Increasing the temperature of heat treatment reduced the remaining PE activity in regular fashion. Unheated concentrates were stable for one week, but all concentrates receiving heat treatments showed complete cloud stability throughout the 6-week test period at 40° F.

TABLE 4

The effect of final Brix in the evaporator on enzyme activity and cloud stability in 58.5° Brix concentrates

Treatment temperature	PE. in juice	Evaporator product		PE. in final product	Activity remaining[1]	Weeks stable at 40° F. (visual)
° F.	PEu./ml. x 10^4 per 12° Brix	° Brix	PEu./ml. x 10^4 per 12° Brix	PEu./ml. x 10^4 per 12° Brix	%	
Control	18.9	62	19.0	19.0	----	1
		64	18.9	21.2	----	1
		66	20.2	20.7	----	1
150	9.6	62	11.0	11.0	54	>6
		64	11.8	10.9	54	>6
		66	11.2	9.3	46	>6
160	2.5	62	4.4	5.5	27	>6
		64	4.5	4.9	24	>6
		66	4.3	4.9	24	>6
170	2.3	62	2.5	4.1	20	>6
		64	2.2	3.0	15	>6
		66	2.1	2.8	14	>6
180	1.8	62	2.0	2.2	11	>6
		64	1.8	2.4	12	>6
		66	2.0	2.8	14	>6

[1] Percent activity remaining calculated from control average of 20.3 PEu./ml. x 10^4 per 12° Brix.

SUMMARY

Frozen concentrates of 42° and 58.5° Brix were prepared from a uniform supply of Valencia oranges. Evaporator feed juices were heat treated to 150°, 160°, 170°, and 180° F., held at the desired temperature for 5 sec., quickly cooled, and concentrated. The cloud stabilities of all 58.5° Brix samples were superior to those of corresponding 42° Brix samples when stored at 40° F. Pectinesterase activity decreased with increased temperature of heat treatment in all cases. Heat treatment at temperatures of 150° F. or higher gave cloud stability in all high-Brix concentrates when stored at 40° F. for 6 weeks. No flavor damage was observed even when treatments of 180° F. were used. Parallel experiments using two different types of heat exchangers did not develop any obvious differences in their effect on stability or flavor. By mixing heated and unheated 68° Brix concentrates and then cutting back to 58.5° Brix with unheated juice it was found that only 50% of the evaporator feed juice needed to be heated to 150° F. to provide cloud stability for 6 weeks at 40° F. When 58.5° Brix products were prepared by cutting back concentrates of 62°, 64°, and 66° Brix, no differences were observed between comparable heat treated samples.

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Some Observations on Red Pepper. I. A New Method for the Determination of Pungency in Red Pepper[a]

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(Manuscript received August 1, 1956)

RED PEPPER IS A MEMBER of the capsicum family which also includes paprika and sweet red pepper. The genus Capsicum contains members which according to Parry *(16)* vary widely in size, shape, flavor, and pungency. The work reported below deals with red pepper, variously called cayenne or chillies, in that the principal use in food is for pungency o· heat. There is considerable confusion in the nomenclature of red peppers, so for this work the term "hot red peppers" will be used and by way of illustration includes such varieties as Domestic Sports, Mombassa Chillies and Nigerian Chillies.

Hot red pepper color. Red peppers are used as a seasoning for two purposes, red color and pungency. There is considerable varietal difference in color and pungency and both attributes vary, depending upon the method of drying and handling as well as subsequent storage. The principal pigment of capsicum is listed by Mayer *(8)* as capsanthin, a carotenoid, $C_{40}H_{58}O_3$. Several closely related carotenoids as well as cis-trans isomers are usually found in conjunction with it. Some of these are capsorubin, zeaxanthin, lutein, kryptoxanthin as well as α and β carotene according to Mayer *(8)*. He lists the following physical properties of capsanthin: m.p. 175-176° C., absorption peaks at 475-500 mμ in petroleum ether (b.p. 70-80° C.) dark red glistening needles soluble in alcohol. Goodwin *(3)* shows the absorption spectrum of capsanthin. He also lists peaks for capsorubin at 543, 505.5 and 470 mμ in carbon bisulfide. Karrer and Jucker *(6)* give detailed information on the isolation, identification and physical constants of capsanthin.

Hot red pepper pungency—capsaicin. Because of the intense activity in carotenoids during the past four decades under such workers as Karrer and Zechmeister, the pigments in hot red peppers have been investigated more fully than the compounds responsible for pungency, the known one being capsaicin.

Newman *(13)* has reviewed the chemistry of capsicum spices. He traces the work from 1816 when Buchholtz showed that the pungent principle could be extracted from red peppers by maceration in the presence of organic solvents. About the turn of the century Thresh first crystallized the pungent principle and named it capsaicin. Several workers tried and failed to establish the structure of capsaicin until Nelson *(11)* in 1910 started his work which culminated in 1919 by his publication of a paper declaring capsaicin to be the amide of vanillylamine and iso-decanoic acid. In answer to critics, Nelson *(12)* was successful in synthesizing capsaicin in 1920 from vanillylamine and the acylchloride of the decanoic acid obtained by degradation from natural capsaicin. He never carried out a full synthesis of the compound, however, and this was left for Späth and Darling *(20)* to complete in 1930 via monoethyladipylchloride to the nonanoic acid to the nonenoic acid with veratrylamine to methyl capsaicin and by saponification to capsaicin.

Capsaicin analysis—chemical. Various colorimetric methods for the determination of capsaicin are reported in the literature. Most of these are based on the color reactions of capsaicin with vanadium oxytrichloride first suggested by von Fodor *(24)*. Tice *(21)* investigated this further and suggested that color be added to approximate the color of the capsicum extract since extracted color had proved a difficulty. Prokhova and Prozorovskaya *(18)* suggested the use of a colorimeter. They also reported that the maximum amount of capsaicin was to be found in the inner walls of the pepper, whereas the hulls and seeds contained virtually no capsaicin. Ting and Barrons *(22)* have used the vanadium colorimetric reaction to distinguish hot peppers from sweet peppers.

Jordan, Rebol, and Thompson *(4)* reviewed the assay history of capsaicin and proposed a standard assay method based on the vanadium oxytrichloride colorimetric reaction. Then Hayden and Jordan *(4)* undertook a lengthy study into the method because it was giving so much difficulty in application, largely due to the interference by extracted colored materials, as well as the reactivity of other non-pungent materials with the vanadium reagent. As a result of this survey, the colorimetric method of Tice was withdrawn from the Pharmacopeia as an official method. Ting and Barrons *(22)* and van Blaricom and Martin *(23)* suggested synthetic color standards, the former based on Malachite green and Naphthol yellow and the latter based on cupric nitrate and potassium dichromate.

Nogrady *(14)* has recently suggested a method for capsaicin based on titration with picric acid using fluorescence disappearance as the end point. A different colorimetric method was suggested by North *(15)* based on measurement of a colored compound produced by reaction of capsaicin with phosphotungstic-phosphomolybdic acid reagent.

Capsaicin analysis—organoleptic. A different path followed in the determination of capsaicin relies on the reactions of human tasters to solutions of pungent red pepper diluted to its threshold of detection to tasters. Micko *(9)* was probably the first to suggest analysis for capsaicin by testing for the burning effect upon the tongue and throat. Nelson *(11)* later took up this procedure and suggested that 0.05 ml. of a solution containing 1 p.p.m. capsaicin represents the limit of detec-

[a] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 12, 1956.

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tion by the organoleptic method. This represents about 0.05 μg. of capsaicin. The present methods for testing are given by Wirth and Gathercoal *(25)* for the USA and in the B.P.C. for Britain. Munch *(10)* has suggested an improvement for the USA method, using piperine as a standard since it is more readily available. There have been attempts through the years to increase the accuracy of this method by the use of statistics and more carefully controlled administering conditions. Fabian *(2)* and Prater *(17)* recently conducted a check sample through many laboratories skilled in this organoleptic analysis and encountered variations up to 150%. The principal difficulties experienced in administering this procedure have been rapid tester fatigue and difficulty in continuing successful tester motivation. Tasters checked for reliability and test level reproducibility lost interest and became resentful or antagonistic on continued sample analysis at the frequency required for operation of a suitable quality control program.

The work reported below was undertaken in an effort to eliminate the difficulty encountered in measuring the pungency of hot red peppers. The organoleptic and vanadyl method had proved impractical and inaccurate in day-to-day operation. The method reported below has proved reliable and reproducible and free of the many pitfalls and difficulties of the above procedures. The work covers the isolation of capsaicin, establishment of its spectrophotometric characteristics, and, finally, development of chromatographic techniques for its separation and measurement in red pepper and red pepper oleoresin.

EXPERIMENTAL

Isolation of capsaicin. Capsaicin was isolated for the subsequent work by a modification of the procedure of Tice *(21)*. The starting material was solvent extracted oleoresin of Mombassa chillies. Two hundred grams of oleoresin were mixed with 400 ml. of n-hexane in a separatory funnel and extracted with three 400-ml. portions of aqueous methanol. The methanol extracts were counter washed with two 400-ml. portions of n-hexane. The hexane fractions were combined and the solvent was evaporated in vacuo to leave 177.9 g. of deep red oil, the non-polar fraction of the oleoresin consisting mainly of fatty material.

The methanol fractions were combined and the alcohol evaporated in vacuo. The residual of this aqueous phase was extracted with 3 x 300 ml. of ether, and washed counter currently with 2 x 150 ml. distilled water. The combined ether extracts were dried over Na_2SO_4 then evaporated to dryness. The residue, the polar fraction, comprised crude capsaicin and weighed 17.6 g. This crude capsaicin was recrystallized three times from ether to yield cure capsaicin. M.P. was 66.0-66.8° C. Pure capsaicin, as could be predicted, is an extremely difficult material to handle because of its strong irritative effect.

Capsaicin analysis—chemical. The absorption spectrum of pure capsaicin exhibits two strong peaks at 280 mμ and 231 mμ, as shown in Figure 1. Of these the one at 280 mμ was selected because it is a sharper peak and this wavelength allowed more freedom in the selection of solvents. Solutions of capsaicin in methanol followed Beer's Law in the relationship between absorbance and capsaicin concentration, see Figure 2. The instrument used in these determinations was a Beckman Recording Spectrophotometer, Model DR.

Solvent extraction of red pepper followed directly by absorbance measurement was found inapplicable because of the presence of materials other than capsaicin which absorbed in the range of 280 mμ. Accordingly, chromatographic techniques were investigated in an effort to eliminate the interfering substances during the final reading. After considerable experimentation the column shown in Figure 3 was adopted. The column is a 1.75 cm. diameter tube packed from bottom up sequentially with basic alumina, acidic alumina, and charcoal. Sodium sulfate is used at the top for high moisture red peppers, since water may lead to interference with charcoal efficiency.

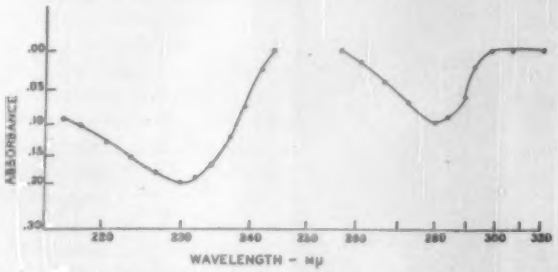

Figure 1. Absorption spectrum of capsaicin.

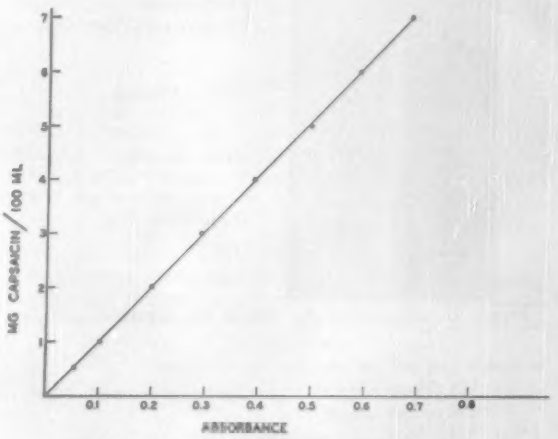

Figure 2. Spectral absorbance at 280 mμ and capsaicin concentration.

Analysis Method:

1. Reagents:
   - Basic alumina (Merck 71707)
   - Acidic alumina (Merck 71695)
   - Charcoal (Norit A-acid washed)
   - Hyflo supercel
   - 1% methanol in ethylacetate (solvent No. 1)
   - Absolute methanol
   - Pure capsaicin—standard, isolated from red pepper
2. Preparation of the column:
   a. Plug tube bottom with small wad of fine glass wool.
   b. Add 12 g. basic aluminum oxide suspended in ethyl acetate containing 1% methanol. Work carefully to be sure column is evenly packed.
   c. In same manner, using same solvent, add 2 g. of acidic alumina.
   d. Add 2 g. of 1:1 mixture of Norit A and filter cell. This is best added at the side to minimize alumina disturbance, use same solvent.
   e. Finally, add a piece of medium filter paper carefully cut to fit the column and top this with 2 g. of anhydrous Na_2SO_4.
   f. Always work under solvent; do not let the column dry.
   g. With the column assembled, pass some solvent No. 1 through the column to purify it, until there is no absorbance at 280 mμ.

Figure 3. Chromotographic column for capsaicin analysis.

3. Sample analysis for oleoresin of red pepper:
   a. Weigh out approximately 200 mg. of oleoresin capsicum and record weight.
   b. Transfer to column using solvent No. 1—about 50 ml. is required.
   c. When oleoresin is on column develop with 250 ml. of solvent No. 1 to wash capsaicin free of interfering substances. At conclusion of washing, allow solvent to just reach column top; do not allow column to emerge from solvent.
   d. Elute capsaicin, using absolute methanol, and collect the eluate in 300 ml. volumetric flask.
   e. Take aliquot of this 300 ml., measure at 280 mμ and calculate using standard curve you have prepared.

4. Sample analysis for ground red pepper:
   a. Suspend 1 g. 20 mesh red pepper in 50 ml. of solvent No. 1 and reflux for 1.5 hours. Cool, and make up to 100 ml. with same solvent. Pipette out 50 ml. aliquot of supernatant liquid.
   b. Transfer quantitatively to column and proceed as above.

Capsaicin analysis—organoleptic. In this work where the chemical method was compared with organoleptic, the Quartermaster method *(1)* for Scoville Heat was used with a slight modification. The Quartermaster method calls for 5% sucrose as oleoresin diluent. It was found that on low heat pepper 3% dextrose did not overshadow "bite" as much as the method of the Quartermaster. Further, in tests conducted triangulation was used with 3% dextrose as blank.

DISCUSSION

Several solvents were tried for this work and it was early apparent that a solvent of low e (dielectric constant) such as ether or ethyl acetate was required for adsorbing capsaicin onto the basic column. This could be followed by a solvent of high e such as acetone or methanol for elution, Lederer and Lederer *(7)*. Several solvents were rejected because of excessive absorbance at 280 mμ—others because of incompatibility with methanol, the eluent of choice. Some produced undesirable turbidity possibly due to moisture in the reagents. Acetone was rejected as eluent because it cuts off the spectral curve before 280 mμ. One percent methanol in ethyl acetate was found to be the best solvent.

In preparation of the column charcoal was used because it absorbs the red pigments and some other interfering substances but passes capsaicin readily in the methanol-ethyl acetate solvent. Of the column materials, charcoal gave the most difficulty. Many types and sizes were tested before the one reported above operated successfully.

The acid-washed alumina is used to separate the charcoal phase from basic alumina. It was found that if these two were in contact there was some capsaicin transfer during elution, and the charcoal clung tenaciously to the capsaicin under these conditions. This did not occur when an acid "spacer" was used. The rate of elution of substances not capsaicin which interfere at 280 mμ is shown in Figure 4. That capsaicin is absorbed

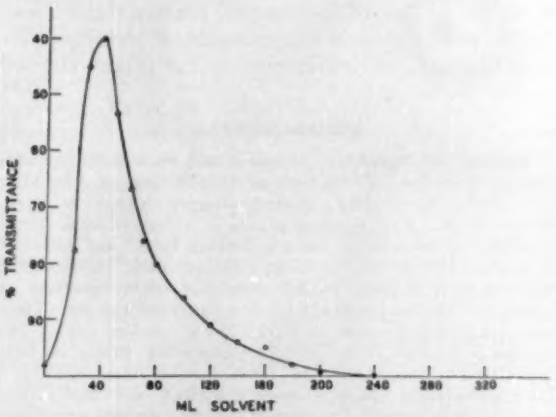

Figure 4. Rate of elution of substances not capsaicin from the chromatographic column.

is demonstrated by the complete lack of pungency in the eluate.

Capsaicin is passed through the column to the basic alumina where it is adsorbed from the ethyl acetate solvent until eluated with absolute methanol. Rate of elution is shown in Figure 5. As shown, capsaicin is recovered by 300 ml. of absolute methanol.

Several samples of different capsaicin contents were run and results are shown in Table 1. Pure capsaicin used as the standard in this work was repeatedly examined for Scoville heat and found to be 15,000,000 Scoville units. This figure was used for analysis of some known types of red pepper (Table 2). The striking heat value of Mombassa and Uganda varieties is shown here, 0.8 and 0.85% capsaicin as contrasted with some Abyssinian chillies at 0.08%.

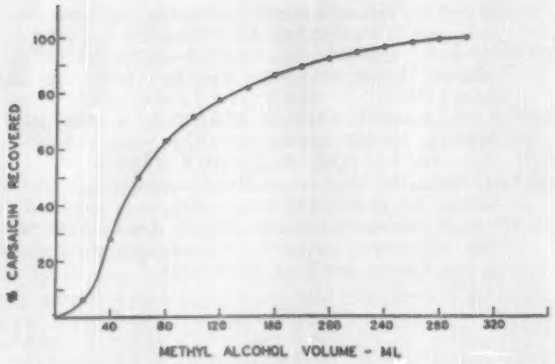

Figure 5. Rate of elution of capsaicin from basic alumina by absolute methanol.

TABLE 1

Results of chemical analysis of typical red pepper oleoresins

Sample no.	Percent capsaicin in oleoresin
	%
1	3.30, 3.30
2	3.18, 3.18
3	3.15, 3.06
4	5.79, 5.79
5	3.67, 3.67
6	5.95, 5.95
7	6.78, 6.78
8	0.76, 0.78
9	0.18, 0.18
10	0.06, 0.059
11	6.78, 6.90

TABLE 2

Capsaicin content of various capsicum fruits

Capsicum type	Scoville heat value	Calculated capsaicin in sample[1]
		%
Hontaka	50,000	.....
Mombassa	120,000	0.8
Santana Chili Pepper	8,400	0.058
Uganda	127,000	0.85
Turkish	12.300	0.083
Mexican Pequinos	40,000	0.26
Abyssinian chillies	11,000	0.075
Japanese Santaka	38,000	0.253

[1] By calculation—using heat value of pure capsaicin as 15,000,000 Scoville units.

TABLE 3

Comparison of analytical results using Scoville organoleptic method and chemical analysis on red pepper oleoresins

Sample no.	Scoville	Chemical[1]
1	425,000	538,000
2	470,000	465,000
3	970,000	876,000
4	285,000	241,000
5	90,000	90,800
6	68,000	51,000
7	345,000	412,500
8	160,000	145,000
9	418,000	477,500
10	154,000	138,000
11	236,000	183,000
12	665,000	540,000
13	230,000	226,000
14	48,000	48,000
15	52,000	40,000
16	48,000	58,000
17	700,000	869,500
18	538,000	553,500
19	845,000	892,000
20	1,025,000	1,035,000
21	157,000	160,000

[1] Reported in Scoville Units Calculated from Capsaicin Content, with Pure Capsaicin as 15,000,000 Scoville Units.

An interesting comparison between Scoville organoleptical pungency and this method is shown in Table 3.

SUMMARY AND CONCLUSIONS

A method is presented for determining the heat or pungency of red pepper using chromatography followed by spectrophotometry of capsaicin. To date it has proved reliable and is quite reproducible. It is difficult to assess this new method in the light of present day organoleptic techniques, since they are subject to gross inherent error not only because of the unreliable test instrument used, but also the rapid fatigue produced and difficulty in achieving satisfactory taste panel motivation.

No difficulty has been encountered in operation of the method and in training new operators if reasonable technical proficiency exists.

Current work indicates this method may well be effective in detecting contamination of black pepper oleoresin by red pepper oleoresin.

Acknowledgments

Grateful acknowledgment is made to the management of William J. Stange Company for their generosity in permitting publication of this work. Acknowledgment is also made to Messrs. Maclyn Peat and Henry Kilar for their assistance in much of the Scoville analytical work.

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2-Thiobarbituric Acid Method for the Measurement of Rancidity In Fishery Products[a]

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(Manuscript received August 3, 1956)

Rancidity is a serious quality problem confronting the fishery industry. The prolonged storage required for many fishery products often leads to deteriorative changes with adverse effects on quality and flavor. Lea *(15)* defined rancidity as any "off odour or flavour which had developed in an oil or fat as a result of deterioration or storage." This report will be concerned with oxidative rancidity or the rancidity caused by the action of air on fat or fatty tissue in fishery products.

The large proportion of highly unsaturated fats which many fish contain partially explains the ease with which fish products undergo oxidative rancidity and develop off-flavors. These off-flavors are described by taste panels as "freezer taste," "cod liver oil flavor," and "rancid taste." Color changes are frequently associated with these off-flavors. These may be seen in the yellowing of frozen fillets and shellfish, and in fading of color in frozen salmon *(7, 13, 24)*. The spontaneous heating of fish meal after it comes from the dryer and the destruction of vitamin A in oxidized fish liver oils are more examples of oxidative rancidity in fishery products.

The stabilization of fat in fishery products by use of antioxidants to prevent oxidative rancidity has been investigated by many workers, *(2, 24)*. The results on frozen fillets have been somewhat contradictory *(1, 8, 16)*, due possibly to the variable composition of the product, difficulty of application of the antioxidant, and the lack of a suitable determinative method for rancidity.

The methods for measuring oxidative rancidity in fats have been discussed in recent reviews by Watts *(26)* and Dugan *(6)*. Marcuse *(16)* states, "There does not exist any reliable, easily applicable, and generally adopted method to measure the products of fat deterioration responsible for rancid taste and flavour. The most used method, i.e., the determination of peroxide value, is used only in default of a better one." Hartman *(10)* recently described a method for the determination of peroxides in fat and phospholipids. He points out that the usual solvents used in the iodometric peroxide method do not extract phospholipids and that these compounds may show rancidity values. The complete extraction of fat from fish meal has been the subject of much study by Stansby *(22)*. It is unlikely that the iodometric procedure would be suitable for rancidity determinations of fish meal samples.

A new method for rancidity evolved when Bernheim *et al. (3)* demonstrated that when certain tissues were incubated with 2-thiobarbituric acid (TBA) the colors produced were a product of the oxidation of unsaturated lipids. Many workers have since applied this reaction to the estimation of rancidity in dairy products *(4, 17, 18, 21)*, frozen pork *(25)*, and fats and oils *(12, 20)*. Fridriksdottir *(9)* used the TBA reaction in a study of the oxidative rancidity of frozen herring. She reported that while the test does distinguish between rancid and non-rancid samples, it was not suitable for the estimation of the degree of rancidity. She also experienced difficulty in obtaining clear solutions for color comparisons and in removal of the color pigment from the fish protein.

The procedure described in this report overcomes the difficulties described by Fridriksdottir. The TBA determination is made on the intact sample eliminating the fat extraction step which is oftentimes difficult, incomplete, and time-consuming. The procedure is simple and gives reproducible results on fish oil, fish meal, and canned and frozen fishery products. The results indicate that it can be used as a measure of oxidative rancidity in fishery products.

EXPERIMENTAL

Reagents. *Trichloroacetic acid solution.* Dissolve 20 g. of trichloroacetic acid, reagent grade, in distilled water and make up to 100 ml.

[a] Approved for publication as Technical Paper No. 978 by Director of Oregon Agricultural Experiment Station as contribution of Seafoods Laboratory of the Department of Food Technology.

Pyridine hydrochloride solution. Mix 30 ml. of pyridine, reagent grade, with 70 ml. of 6 N hydrochloric acid.

Note: Some lots of pyridine give high blank values. Purify by adding 1 gram of 2-thiobarbituric acid per 100 ml. of pyridine, reflux 1 hour, distill.

Alternate: Dissolve 43 g. of pyridine hydrochloride (Eastman grade) in distilled water, add 8 ml. of 6 N hydrochloric acid and make up to 100 ml.

2-Thiobarbituric acid (TBA) reagent. Prepared according to Kohn and Liversedge *(14)* with the exception that the charcoal treatment was omitted. The 2-thiobarbituric acid was an Eastman product.

Hydrochloric acid, trichloroacetic acid, pyridine reagent. Mix 650 ml. of 0.6 N hydrochloric acid with 50 ml. of trichloroacetic acid solution, and add 50 ml. of pyridine hydrochloride solution.

Petroleum ether. Skellysolve F.

Apparatus. Flat bottom boiling flask, 250 ml. short neck, 24/40 ST.

Condenser, 400 mm. jacket West type, 24/40 ST.

Water bath.

Centrifuge tubes, 50 ml.

Centrifuge.

Separatory-centrifuge tubes, Hennessy, from E. Machlett & Son, N. Y. 10, N. Y.

Klett-Summerson photoelectric colorimeter or Beckman DU spectrophotometer.

Treatment of samples. Fish meals: Meals were prepared from black rockfish *(Scorpaenidae sp..)* scrap and whole flounder *(Platichthys stellatus)* by steaming the raw fish for 30 minutes and then passing through a grinder. The ground fish was pressed in a Carver press to eliminate as much liquid as possible. The press cakes were ground and dried in a vacuum oven at 100° C. for 18 hours. The dry meals were ground in a hammer mill. Fresh, frozen, or canned fish: Fresh fish or scrap were put through a grinder while fish fillets and canned products were disintegrated in a Waring blender.

Procedure. Accurately weigh 0.220-0.260 g. of fish meal in a 250 ml. short neck flat bottom boiling flask with ground glass joint. Add 4 ml. distilled water, 5 ml. pyridine hydrochloride solution, 10 ml. trichloroacetic acid solution and 6 ml. TBA solution. Avoid shaking the flask. Connect with a condenser and place the flask in a vigorously boiling water bath and start timing as soon as the flask is placed in the water bath. Reflux, and in exactly 30 minutes, add through the top of condenser 75 ml. of hydrochloric, trichloroacetic acid, pyridine reagent. Shake the flask several times and continue refluxing for 10 minutes. Cool the flask to room temperature in a water bath and disconnect the condenser. The color varies from pink to deep red depending upon the degree of rancidity of the meals. Centrifuge about 40 ml. of the solution in a 50 ml. centrifuge tube for 5 minutes at 1800 r.p.m. Measure 15 ml. of the clear solution in a Hennessy tube, add 10 ml. petroleum ether and shake vigorously for ½ minute *(18)*. Centrifuge 3 minutes at 1200 r.p.m. The solution should be crystal clear. In case of any cloudiness of the solution, shaking with petroleum ether should be repeated. Draw the clear solution into a colorimeter tube or 1 cm. Beckman corex cuvet. Color density is determined in a Klett-Summerson photoelectric colorimeter using a No. 54 filter or with a Beckman DU spectrophotometer at 535 mμ. The reagent blank is checked occasionally and was found to give values of 6 to 7 in a Klett colorimeter tube checked against a distilled water blank. The results may be expressed directly in Klett reading if the sample size is constant or preferably in $E^{1\%}_{1cm}$ at 535 mμ, using a Beckman spectrophotometer.

For canned, fresh, or frozen fish samples, weigh 0.900 to 1.100 g. sample in the flask, add 3 ml. of distilled water and disperse the sample in the water with a stirring rod before adding the other reagents. This will avoid lumping of the sample. The stirring rod is washed free of any adhering sample with 10 ml. of trichloroacetic acid solution. The rest of the procedure follows exactly as detailed in the above paragraph for fish meals.

A sample size of 0.220 to 0.260 is used for fish oils and the procedure follows as described for fish meal.

RESULTS AND DISCUSSION

Absorption spectra. When oxidized fish products reacted with the TBA reagent, a color ranging from light pink to deep red resulted. Fresh fish materials gave little or no color. The absorption spectra of the color produced with turbot *(Atheresthes stomias)* and rockfish meal is shown in Figure 1. The peak of absorption is at 535 mμ, which agrees with data reported for rancid milk fat *(17)*, rancid pork *(25)*, frozen herring *(9)*, oxidized linseed oil *(3)*, and malonic dialdehyde *(11, 17, 18)*.

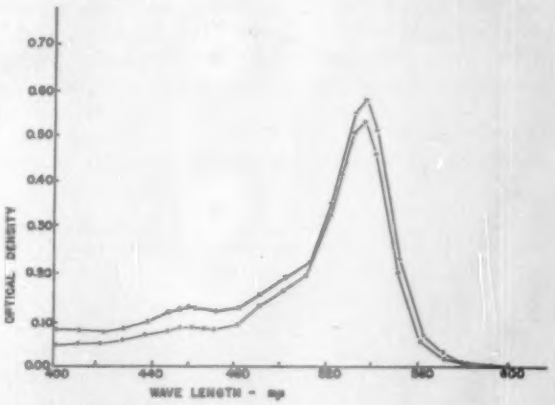

Figure 1. The absorption spectra of the colors produced with TBA by oxidized fish meal samples, Turbot meal (o) and Rockfish meal (x).

A slight peak in the absorption spectra was observed at 458 mμ with many fish products. Fridriksdottir *(9)* reported a similar peak in frozen herring at 454 mμ. Bernheim *et al. (3)* showed a similar peak with certain oxidized lipids and TBA. Wilbur *et al. (27)* reported that irradiated mouse skin gave two maxima on reaction with TBA reagent, a main peak at 535 and a lesser one at 450-460 mμ. In our studies, a sample of commercial meat meal gave a yellow color under these conditions and it was found that the maximas at 458 and 535 mμ gave an optical density of 0.146 and 0.080, respectively. The interference by certain carbohydrates has been studied by several workers *(5, 25, 27)*. However, in products composed entirely of fish very little interference has been noticed.

Reaction times and conditions. The density of the color produced with rancid fish materials and the TBA reagent is related to the reflux time. Figure 2 shows that the rate of increase of color was greatest during the first 30 minutes of refluxing. From there on the increase was slower but continuous. The total time of heating for the determination was 40 minutes. This was found to give excellent replicate results. Eleven replicate determinations were made upon a single sample of oxidized rockfish meal to determine the reliability of the method The arithmetic mean was found to be 0.406. The standard deviation from the mean was 0.002. The standard error of the mean was 0.001. The 99% confidence limits for the true mean of this sample was 0.403 to 0.409.

The reaction was performed under a reflux condenser to maintain a more constant volume. We observed in the more rancid samples that a small quantity of volatile material, capable of reaction with the TBA reagent, would be trapped by the condenser. Washing

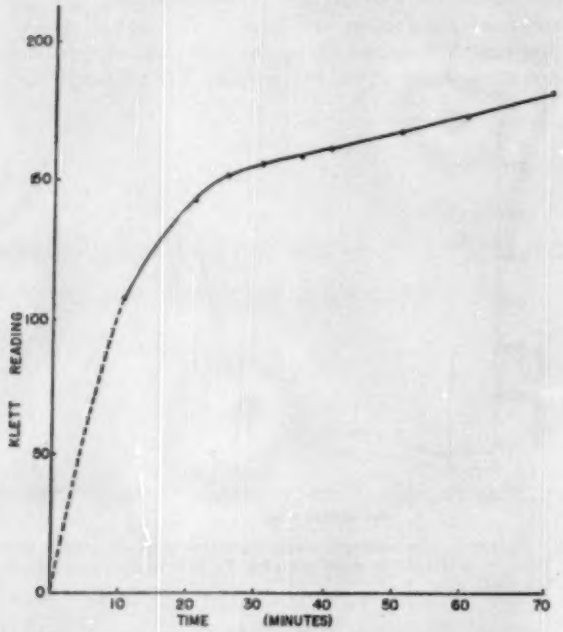

Figure 2. The effect of time of heating with TBA reagent at 100° C. upon intensity of color, rockfish meal.

down the condenser with reagent corrected this condition.

Fridriksdottir *(9)* experienced difficulty in removing the red color from the fish protein and in obtaining clear solutions for color comparisons. Boiling the solution with additional reagent facilitated the removal of color from the protein and sufficiently diluted the colored solution for color measurement. Trichloroacetic acid was effective in maintaining a clear solution. The use of pyridine hydrochloride reagent in the reaction and washing mixture yielded higher results and seemed to extract the color from the meal. This is shown in Table 1. The use of Roccal recommended by Briggs and

TABLE 1

Effect of pyridine hydrochloride on color density

Sample	Color density—Klett reading	
	with pyridine HCl	without but with equivalent HCl to give the same pH
Commercial tuna meal	159	146
Rockfish meal	510	453
Salmon oil	320	315

Bryant *(4)* did not effect the reaction under the conditions described. Patton and Kurtz *(18)* eliminated any slight turbidity in the final solution by shaking with petroleum ether; our work confirmed their observation.

Extraction of rancid products from fish meal. Rancidity determinations of fish meal fat is an imposing problem because of the difficulty of complete fat extraction. Stansby *(22)* in his studies leading to a tentative A.O.A.C. method for fat in fish meal found that, in stored fish meal, ethyl ether extraction gave less than 50% of the true fat value. He also reported that the binary azetotropic mixture of methanol-benzene gave a more complete extraction of fat than the other solvents tested *(23)*. Hartman *(10)* employed a mixture of methanol-benzene as a solvent for lipids in his peroxide method. His data showed higher peroxide values which he attributed to the phospholipid fraction which was extracted by methanol-benzene. These studies stressed the difficulty of solvent extraction of oxidized fat and rancid products. For this reason it appeared that a rancidity procedure suitable for fish meal would be readily adaptable to other fish products. This proved to be the case. The TBA procedure was conducted on the intact sample and the difficulty of solvent extraction eliminated.

However, because of the reported interference by carbohydrate *(4, 5, 25, 27)* it would be of interest to determine the efficacy of a solvent procedure. Such a modification would be of value in rancidity determinations of mixed feeds as well as dog and cat foods, many of which are high in carbohydrate. The methanol-benzene procedure of Hartman *(10)* and the ethyl ether-petroleum ether method described by Risley *(19)* were selected. Acetone extraction was also attempted. Oxidized rockfish meal was used as the sample. The results are shown in Table 2.

TABLE 2

Extraction of TBA reactive material[1]

Solvent	Klett reading		
	Intact meal	Solvent extract	Residue
Methanol-benzene	137	22	117
Ethyl ether-pet. ether	131	25	120
Acetone	128	18	129

[1] The determinations were made on a 0.25 g. sample of the intact meal, and the total extractables and residue from 0.25 g. of original meal.

These results show that the solvents tested are not suitable for extracting the material that reacts with the TBA reagent. The determination should be performed on the intact fish meal. These data suggest that the very active substances, malonic dialdehyde, which is found on oxidation of fat has reacted with protein or itself to yield a compound that is insoluble in the solvents tested. Additional work is in progress regarding this point.

TBA value of fishery products during storage. Vacuum dried flounder, carefully prepared from very fresh fish, gave an initial value of 44 (0.25 g. sample) using a Klett colorimeter. The meal was stored at room temperature (22° C.) in an open glass jar and periodically tested for TBA value and odor change. By the seventh day a distinctly rancid odor was evident, Klett reading 160. The TBA value increased and reached its highest level, Klett value of 187, after 9 days of storage. Then it dropped to a fairly constant value of 150 as shown in Figure 3.

Flounder, ground while still in rigor, was stored in a bottle at 2° C. A gradual increase was noticed in TBA,

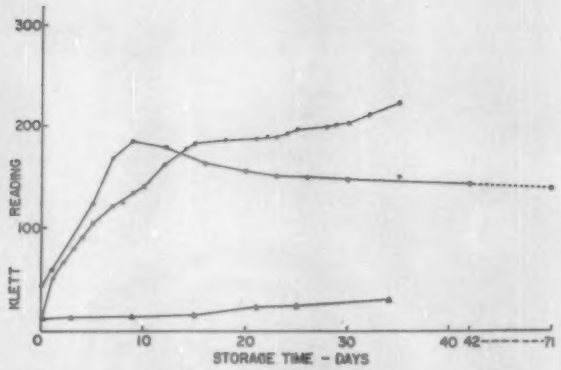

Figure 3. The relation of TBA values to storage time.
Flounder meal, 22° C. (o)
Flounder whole, ground, 2° C. (x)
Flounder, whole, then filleted, 2° C. (△)

Figure 3. A fishy off-odor developed in 15 days. In contrast, whole flounder from the same group of fish showed little change from the initial TBA value. The storage temperature was 2° C. and fillets were cut from two fish for each test.

It would appear from the results shown in Figure 3 that the TBA test could be applied to storage tests of fish meal and ground fish.

TBA values of fishery products. Table 3 shows the TBA values of a variety of fishery products. There appears to be a relationship between taste and TBA values for many products. The suggested procedure was satisfactory for all samples except those containing large amounts of carbohydrate, i.e., dog food, cat food, etc.

Fat determinations were made by drying the wet samples with anhydrous sodium sulphate and extracting

TABLE 3
TBA value of commercial fishery products

Sample	Grade	Organoleptic rating	Fat	Free fatty acids	TBA value	Remarks
Canned:			%	%	$E^{1\%}_{1\,cm.}$	
Salmon						
O. tshawytscha	fancy	good	10.9	0.77	0.047	1955 pack
	fancy	poor, metallic	26.7	1.19	0.527	14 years old
O. kisutch	fancy	good	.....	.....	0.075	1954 pack
Tuna						
T. germo	solid	good	25.3	0.45	0.044	1955 pack, all fish of same lot
	chunk	good	22.8	.....	0.056	
	grated	good	24.7	0.57	0.070	
N. macropterus	solid	good	23.0	0.49	0.053	1955 pack
	chunk	good	22.5	0.50	0.046	1955 pack
	solid	fair	19.9	0.47	0.110	.....
Herring						
C. harengus	standard	fair, sharp	20.6	1.10	0.092	Eastern U. S. pack
Anchovy						
E. mordax	standard	fair	10.9	0.103	0.100	Western U. S. pack
Mackerel						
P. diego	standard	fair	6.34	0.533	0.064	Western U. S. pack
Frozen:						
Smelt						
T. pacificus	eviscerated	fair	12.6	.....	0.062	.....
Rockfish						
Sebastodes sp.	fillets	poor	2.14	0.53	0.107	entire fillet
	fillets	very poor	.....	.....	0.600	surface of above fillet
	mink food	rancid	.....	.....	0.307	entire fish, 2 mos. in freezer
Halibut						
H. stenolepis	steaks	good	.....	.....	0.030	.....
Sole						
M. pacificus	fillets	fair	.....	.....	0.063	.....
Salmon						
Onchorhynchus sp.	steaks	poor	.....	.....	0.227	.....
Tuna						
N. macropterus	precooked	good	3.26	0.26	0.056	before canning
T. germo	precooked	good	.....	.....	0.037	before canning
Flounder						
P. stellatus	fresh	good	.....	.....	0.015	fish dead one hour
Fish Meal:						
Rockfish						
Sebastodes sp.	.....	oxidized	8.19	.....	1.56	commercial sample
	.....	good	7.00	.....	0.459	laboratory prepared, fresh
	.....	oxidized	7.00	.....	6.50	laboratory prepared, old
Dover sole						
M. pacificus	.....	oxidized	27.9	.....	1.30	laboratory prepared, old
Tuna						
T. germo	.....	oxidized	7.97	.....	1.88	commercial meal
Bonito						
Sarda sp.	.....	oxidized	.....	.....	6.32	fresh meal, frozen fish
Blueback whale						
S. musculus	.....	oxidized	13.3	.....	3.80	meat frozen, 3 years
Herring						
C. pallasii	.....	oxidized	5.73	.....	2.30	commercial
Flounder						
P. stellatus	.....	oxidized	1.47	.....	1.76	laboratory prepared, old
Fish oil:						
Salmon						
O. tshawytscha	edible	poor	.....	0.570	2.73	old sample
	edible	poor	.....	0.866	2.54	old sample
	edible	good	.....	0.171	0.320	new sample
Sardine						
S. caerulea	commercial	very poor	.....	.....	6.32	old sample
Menhaden						
Brevoortia sp.	commercial	good	.....	.....	1.02	new sample
	commercial	good	.....	.....	1.09	alkali processed

the sample in a Waring blender with petroleum ether. All the extracts were combined in a 250 ml. volumetric flask. A portion was centrifuged until clear and a suitable aliquot removed and evaporated on a steam bath under a stream of nitrogen. The residue was weighed and the fat calculated. Fish meal samples were assayed for fat content by the identical procedure on the day of preparation. Old fish meal samples were assayed for fat content by the method described by Stansby *(23)*. Free fatty acid determinations were run by dissolving the fat in a neutralized solution of isopropanol and titrating with 0.1N-NaOH in an atmosphere of nitrogen using thymolphthalein as in indicator.

SUMMARY

The modified TBA method described in this paper was found to be an effective means for the determination of oxidative rancidity in a wide variety of fishery products. The oxidation of fat in fish meal, fish oil, fresh, and frozen fish was satisfactorily determined by this procedure. The reaction is performed on the intact sample eliminating the difficult extraction which is necessary in many rancidity methods. Conditions which affect the sensitivity of the reaction were investigated. Preliminary results indicate that it may be used as an index of quality of stored fishery products. Additional studies are in progress.

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The Time-Temperature Tolerance of Frozen Foods. IV. Objective Tests To Measure Adverse Changes In Frozen Vegetables

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THE NUMBER OF ARTICLES PUBLISHED in the past 25 years on objective tests for measuring quality of frozen vegetables indicates the importance of this field of research. In 1937, Campbell *(1)* noted that undesirable color changes occurred in frozen peas when stored at temperatures above 0° F. and demonstrated that they were due to conversion of chlorophyll to pheophytin. Mackinney and Weast *(8)* proposed the use of chlorophyll and its degradation product, pheophytin, for measuring adverse color changes in frozen vegetables and described a method for determining the ratios of concentration of these two compounds. Kaloyereas *(5)* has suggested that the amount of liquid draining from fruits and vegetables after thawing is related to the quality of the product. Woodroof and Shelor *(16)* noted that the size of ice crystals in frozen products is correlated with freezing conditions and post-freezing storage history. Lee and Wagenknecht *(6)* stored unblanched peas at 0° F. for 5 years and showed that the peroxide number, iodine number, and acid number of the extracted lipid material changed during storage and that rancid off-flavors developed in the peas. Tressler and Pederson *(13)* suggested ascorbic acid and the number of micro-organisms present in frozen foods as indices of quality. Joslyn and David *(4)* found that raw or underblanched peas accumulated alcohol and acetaldehyde during frozen storage, which paralleled off-flavor development. Wagenknecht *et al. (15)* followed the loss of chlorophyll during storage of unblanched frozen peas and postulated that lipoxidase is involved in the destruction of chlorophyll. Total ascorbic acid and its degradation products—dehydroascorbic and diketogulonic acids—have been shown to remain constant in orange juice during storage for 36 days at 36° F. by Mills *et al. (10)*. No mention is made of using the ratios of reduced ascorbic acid to its oxidation products as an index of quality for frozen foods in the latter article, but the results obtained indicate that this may provide a useful tool for assay of quality, provided the sum of reduced ascorbic acid and its oxidation products remains constant in other foods as was demonstrated in orange juice. Other ways of determining quality of frozen vegetables have been proposed, including presence of cavity ice, ratios of chlorophylls *a* and *b*, and amount and type of organic acids and amines.

Evaluation of these tests and development of new tests for quality of frozen foods have been considered an important part of the comprehensive studies of changes in frozen foods which are presently under way in this Branch. The objectives and general plan for these studies have been described by Van Arsdel *(14)* in the introductory article of this series on the "time-temperature tolerance of frozen foods." Extensive subjective appraisal of samples under test by trained judges is an integral and important part of the work, in order to correlate changes in color, flavor, and texture obtained by objective measurements with those determined organoleptically.

In this work, a number of lots of frozen vegetables were obtained from various packers. These were selected to be representative of principal producing areas, leading commercial varieties, and processing variables encountered in the several plants. These various lots were shipped with dry ice to Albany, California, and subjected to various constant temperatures, fluctuating temperatures, and time-temperature cycles simulating those encountered in handling frozen foods. At intervals, samples were withdrawn from the various sublots undergoing test for objective and subjective appraisal.

Most of the program on frozen peas and green beans has been completed and work on cauliflower and spinach is in progress. Other frozen vegetables will be added to the program later. Data obtained by subjective appraisal and the correlation of subjective and objective test results will be reported in a later paper.

METHODS AND MATERIALS

The large number of samples involved in these tests made it mandatory to limit the number of objective tests that could be carried through the program. Since these commercial samples were all adequately blanched, acetaldehyde, alcohol, and changes in lipid material, which occur only in raw or underblanched material, were eliminated from the program as quality indices after some preliminary experiments. Test for adequacy of blanch was by the Masure-Campbell *(9)* peroxidase test on a filtered extract. Ice-crystal characteristics were difficult to measure and define and the amount of liquid draining from thawed samples did not seem to be correlated with changes during storage. Changes in the absolute amount of chlorophyll or in the ratio of chlorophylls *a* and *b* did not readily provide as much information as obtainable from the ratio of chlorophyll to pheophytin.

Four objective tests have been used to date: (1) reduced ascorbic acid, (2) total reduced ascorbic acid and its oxidation products, (3) percent chlorophyll converted to pheophytin, and (4) changes in reflectance color as measured by the Hunter Color and Color-Difference Meter.

Reduced ascorbic acid and its oxidation products. Reduced ascorbic acid, dehydroascorbic acid, and diketogulonic acid were determined by the procedure of Mills *et al. (10)*. In this procedure, the filtrate from a slurry of vegetable acidified with metaphosphoric acid is treated with H_2S gas to reduce dehydroascorbic acid (DHA) to reduced ascorbic acid (AsA), and CO_2 is bubbled through to remove excess H_2S. Diketogulonic acid (DKA) is obtained by direct coupling with 2,4-dinitrophenylhydrazine (DNPH). Thiourea is added to prevent oxidation of AsA. DHA and DKA are determined on a second portion of filtrate by coupling with DNPH. Total AsA, DHA, and DKA are determined on a third portion of filtrate by oxidizing reduced

AsA with a slight excess of bromine and coupling with DNPH after removing excess bromine by aeration. By proper subtractions, the amount of each compound is obtained. Reduced ascorbic acid determined by this procedure was found to be 2 to 5% higher than that obtained by the indophenol method as described by Loeffler and Ponting *(7)* as modified by Ponting *(11)*.

Chlorophyll-pheophytin. A modification of the method of Dutton *et al. (3)* to provide for a larger sample was used to determine the percent conversion of chlorophyll to pheophytin. An 80% acetone extract of frozen vegetable was made by the procedure described by Dietrich *et al. (2)*. The optical density of the filtered extract was measured at 534 and 556 mμ with a spectrophotometer and the ratio of chlorophyll to pheophytin determined by the method of Mackinney and Weast *(8)*.

Reflectance color. Color attributes of samples of peas and green beans were measured with a Hunter Color and Color-Difference Meter *(2)* set to a reference tile with values of *Rd*, *a*, and *b* of 31.0, —24.0, and +13.1, respectively. The frozen material was thawed in the package 3 hours at room temperature. Three readings were made on material from one or two packages. The material was mixed between readings. Samples were submersed in 0.5 to 1.0 inch of distilled water in a cell 3.3 inches in diameter and reflected light readings for values of *Rd*, *a*, and *b* were taken through a 2.25-inch circular opening in the cover plate.

The following lots of raw material, consisting of 50 to 100 dozen retail packages per lot, were used in the experiments: *Peas:* 4 lots, 3 varieties, 3 growing areas, 3 crop years. *Green snap beans:* 5 lots, 4 varieties, 3 growing areas, 3 crop years. *Cauliflower:* 2 lots, 2 varieties, one growing area, one crop year.

RESULTS

Ascorbic acid and related products. Changes in reduced ascorbic acid during storage at 20° F. are shown in Figure 1 for several lots of peas, green beans, and cauliflower. Differences in initial reduced ascorbic acid contents between the different vegetables and lesser differences between varieties of the same vegetable are readily apparent. Differences in rates of loss are clearly indicated by the slopes of the several lines. In Figure 2, losses of reduced ascorbic acid in peas stored at 0°, 10°, 20°, and 30° F. are shown.

Results of analyses for the sum of AsA, DHA, and DKA in several lots of peas, green beans, and cauliflower stored at several temperatures are shown in Figure 3. In the samples of green beans stored at 10°, 20°, and 25° F., the total amount of reduced ascorbic acid and its oxidation products remained almost constant. AsA, which is being oxidized under these conditions, is being converted into DHA and DKA with the reaction proceeding no further. Since there are marked decreases in the AsA contents of the two samples of cauliflower and a lesser decrease in the one sample of peas, it is obvious that reaction products other than DHA and DKA (perhaps involving fragmentation of the DKA molecule) are being produced.

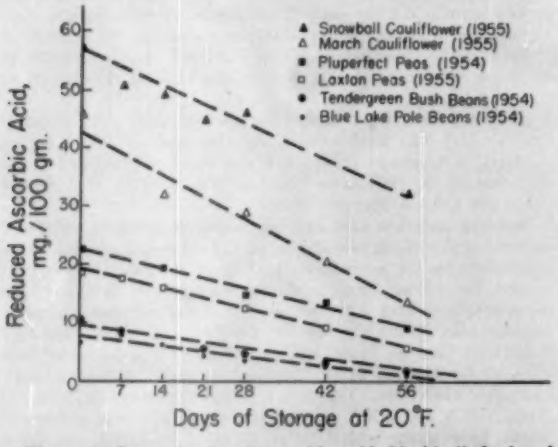
Reduced Ascorbic Acid, mg./100 gm.
Days of Storage at 20°F.
▲ Snowball Cauliflower (1955)
△ March Cauliflower (1955)
● Pluperfect Peas (1954)
□ Laxton Peas (1955)
● Tendergreen Bush Beans (1954)
• Blue Lake Pole Beans (1954)

Figure 1. Loss of reduced ascorbic acid (AsA) (indophenol method) in cauliflower, peas and green beans at 20° F.

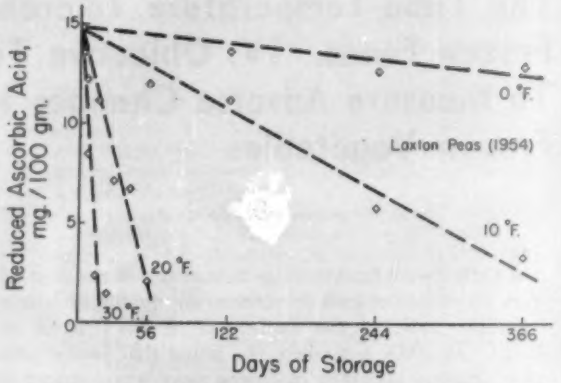

Figure 2. Loss of reduced ascorbic acid (AsA) (indophenol method) in Laxton peas (1954) at 0°, 10°, 20°, and 30° F.

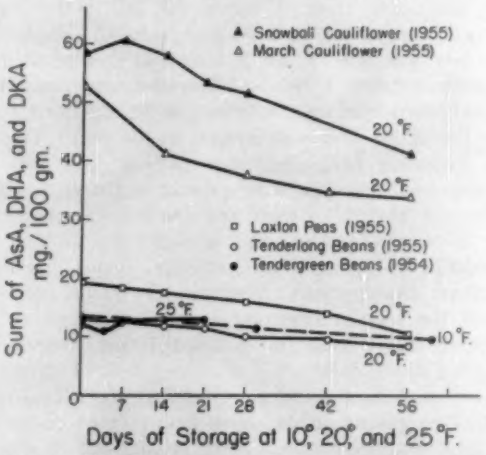

Figure 3. Effect of storage on the sums of reduced ascorbic acid, dehydroascorbic acid, and diketogulonic acid in cauliflower, peas, and bush beans at 20° F.; and at 10° and 25° F. for bush beans.

Amounts of AsA, DHA, and DKA found in one lot of Tendergreen beans stored at 25° F. are plotted in Figure 4. In this experiment, the sum of the 3 compounds remained almost constant, AsA decreased markedly, and the DHA and DKA increased by substantial amounts. Similar data for peas and cauliflower are shown in Figures 5 and 6. In both peas and cauliflower, AsA and sum of AsA, DHA, and DKA dropped sharply. DHA remained almost unchanged and fairly large increases in DKA occurred. It will be noted that initially appreciable amounts of DHA and DKA were found in the various samples. This is not surprising, since Mills *et al. (10)* found several mg./100 g. of DHA and lesser amounts of DKA in a number of "fresh" fruits and vegetables, particularly after holding at room

temperature. Rates of accumulation of DKA in green beans, cauliflower, and peas stored at 20° F. for periods up to 8 weeks are shown in Figure 7.

Conversion of chlorophyll to pheophytin. Data obtained for conversion of chlorophyll to pheophytin in several lots of peas and beans are plotted in Figure 8. These data show that chlorophyll in peas is considerably more stable than chlorophyll in beans during processing and storage. The amount of converted chlorophyll found initially is assumed to have occurred during processing, since Mackinney and Weast *(8)* and unpublished experiments in this Branch have shown that little or no pheophytin is present in freshly harvested peas or beans. It is interesting and perhaps significant from a commercial viewpoint that the rate of change of chlorophyll to pheophytin in beans proceeds more rapidly if processing has been conducted in such a way as to cause excessive chlorophyll degradation. Figure 9 illustrates the effect of storage temperature on chlorophyll degradation in green beans at 0°, 10°, 20°, and 30° F.

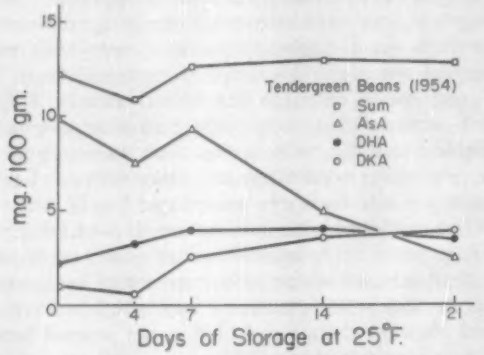

Figure 4. Effect of storage at 25° F. on the AsA, DHA, DKA content of (1954) Tendergreen bush beans graphed separately and as a sum of the three compounds.

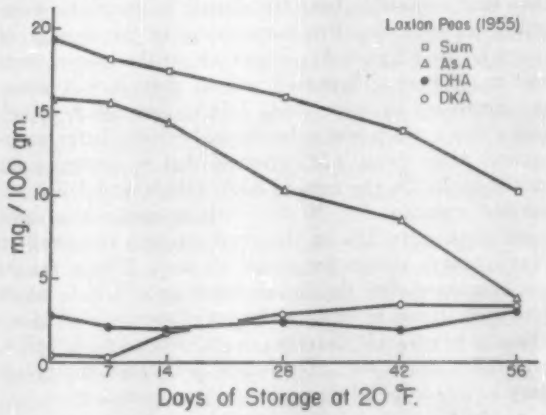

Figure 5. Effect of storage at 20° F. on AsA, DHA, DKA and the sum of these compounds in (1955) Laxton peas.

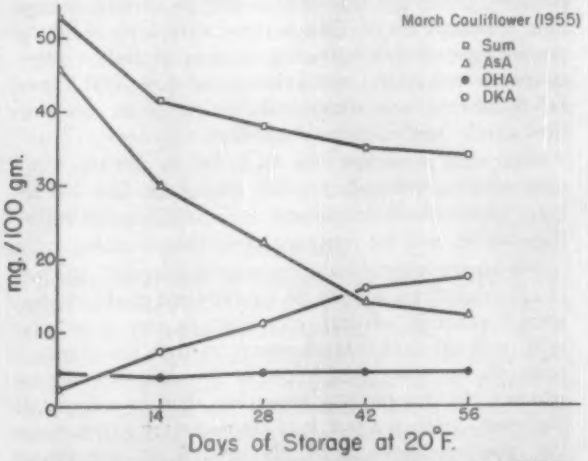

Figure 6. Effect of storage at 20° F. on AsA, DHA, DKA, and the sum of these compounds in (1955) March cauliflower.

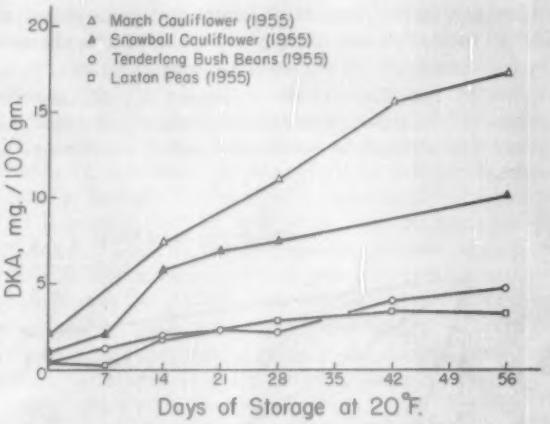

Figure 7. Effect of storage at 20° F. on the 2,3-diketogulonic acid (DKA) content of cauliflower, bush beans, and peas.

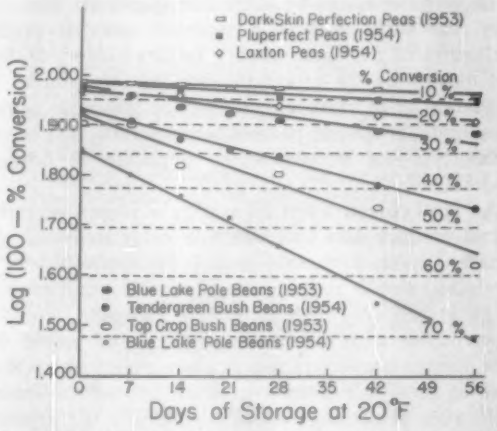

Figure 8. Effect of storage on the percentage of conversion of chlorophyll to pheophytin in peas and green beans at 20° F.

Reflectance color. Color changes during storage of frozen green beans and peas, as measured by the Color-Difference Meter, differ markedly. The ratio of a/b, which shows a change in hue from green towards yellow, best represents changes in color of green beans. The quantity $\sqrt{a^2 + b^2}$, which is a measure of change in saturation, best represents changes in peas. A decrease in the latter value indicates a grayer, duller, less vivid color. Robinson *et al. (12)* have illustrated and discussed the use of these calculations to express color in tomato juice and their conversion to other color systems.

Data obtained from bush beans and peas stored at 20° F. have been selected to illustrate respective changes in a/b and $\sqrt{a^2 + b^2}$. It is practicable to plot these values in the form shown in Figure 10, because the values fall in a favorable area in Hunter's chromaticity graph and changes in color cover only a small range of values.

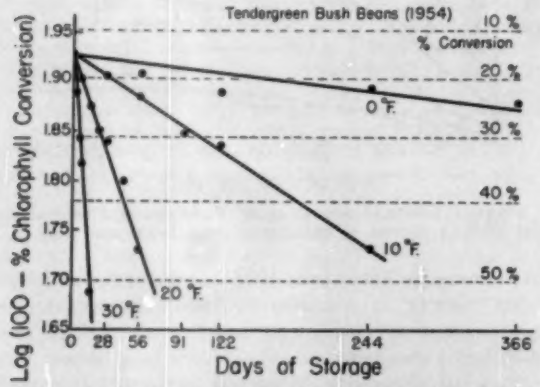

Figure 9. Increase in the percentage of conversion of chlorophyll to pheophytin in bush beans at 0°, 10°, 20°, and 30° F.

DISCUSSION

Determination of quality of frozen foods can be made by objective tests and by subjective appraisal. Theoretically, the latter method is superior, since it permits evaluation of the same quality factors—color, texture, and flavor—by the procedure that will be used by the consumer. Unfortunately, subjective appraisal is time consuming, is subject to considerable error, and is very difficult, if not impossible, to duplicate in different laboratories.

An ideal objective test for quality of frozen vegetables would produce data that would be correlated with subjective appraisal results, would be applicable to all products, would require no expensive equipment or reagents and only a short time to complete, would be reproducible with different operators, and would provide definite answers from a single sample without reference to condition of raw material or product immediately after processing, which normally is not available. It is unreasonable to expect any objective test to meet this ideal; however, the knowledge that the tests reveal is important in the current industrial effort to reduce changes in quality through control of temperature and other environmental factors.

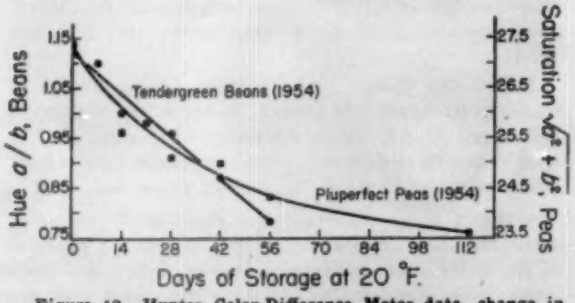

Figure 10. Hunter Color-Difference Meter data, change in hue (a/b) and saturation ($\sqrt{a^2 + b^2}$) for bush beans and peas, respectively, at 20° F. storage.

Decreases in amounts of AsA are correlated well with time-temperature history for frozen vegetables (Figures 1 and 2) and parallel changes in color as measured by change of chlorophyll to pheophytin. Data from this Branch (to be published soon) show that changes in AsA content are also closely related to flavor and color as determined by subjective appraisal. There is, however, one very serious shortcoming: determinations made on a single sample have very little value alone and are useful for determining temperature history only when retention can be calculated. This of course requires that AsA content after processing be available, since this value varies with variety, growing area, processing conditions, and other factors. Underblanching is also known to cause rapid loss of AsA.

When combined with data on amounts of DHA and DKA present, AsA becomes much more useful as a quality index and as an indicator of time-temperature history. Maximum usefulness and reliability are obtained when the change of AsA does not proceed beyond DKA or, to state this another way, while the sum of AsA, DHA, and DKA remains constant. Under these conditions, there is little need for information other than that obtainable from the sample in question. Conditions approaching this were found in the storage of green beans (Figure 4). However, in the case of peas and cauliflower (Figures 5 and 6) there are substantial decreases in total AsA, DHA, and DKA which make this a much less valuable tool for the latter vegetables. Mills *et al. (10)* showed that in orange juice stored at 36° F. the sum of AsA, DHA, and DKA remained constant for 30 days while, under the same conditions, only 5% of the total amount remained in potato slurry stored for about 19 days. These results are consistent with the known behavior of DKA, which has been shown to undergo fragmentation of its carbon chain at pH 6.7. Differences in stability of AsA, DHA, and DKA noted for green beans, peas, and cauliflower may be due to pH differences of these commodities.

In all lots of vegetables that have been investigated to date, there has been a gradual increase of DKA proportional to storage temperature and paralleling storage time reasonably well. Much more data are needed on the increase of DKA during storage of frozen vegetables, as well as on overall changes of AsA, DHA, and DKA, before these compounds, singly or in combination, can be useful as quality indices.

Analytical procedures for AsA, DHA, and DKA are somewhat complicated and time consuming and will require considerable refinement and simplification before they can be used for purposes other than research.

For frozen vegetables containing chlorophyll, the loss of chlorophyll appears to be an excellent quality index, since it parallels adverse color changes, loss of ascorbic acid, and off-flavor development. Other investigators have shown that pheophytin is the sole degradation product of chlorophyll in vegetables that are adequately blanched. In such a test, it is not necessary to determine the absolute amount of chlorophyll or pheophytin present, since all of the required information can be

obtained from the ratios of these two compounds. This procedure is straightforward, reproducible, and takes only moderate time.

Interpretation of data is complicated somewhat, particularly for green beans, where substantial conversion of chlorophyll to pheophytin occurs during processing (Figures 8 and 9). In the samples tested to date, this conversion ranged from 7 to 30% for green beans and from 3 to 8% for peas. However, since color is one of the most important factors in determining quality, color deterioration, whether during processing or storage, is equally limiting in terms of consumer acceptance.

Differences in stability of chlorophyll between green beans and peas are quite striking (Figure 8). It is known that the conversion of chlorophyll to pheophytin —the only chlorophyll reaction that occurs in adequately blanched frozen vegetables—involves the exchange of magnesium of the chlorophyll molecule for hydrogen ion and that this reaction is favored by lower pH. The lower pH of green beans is quite likely one of the factors responsible for greater losses of chlorophyll in green beans.

Hue *(a/b)*, saturation ($\sqrt{a^2 + b^2}$), and lightness *(L)* of frozen beans and peas as measured with the Hunter Color and Color-Difference Meter were all affected by storage. Peas and beans darkened rapidly (*L* value decreased) early in storage and then remained almost unchanged. Saturation values for peas decreased with increasing storage time while hue remained nearly unchanged in the early stages of storage and then decreased rapidly. Saturation and hue of green beans changed progressively with increasing storage, but changes in hue were considerably more consistent than changes in saturation.

Reflectance measurements on frozen peas and green beans fusnish at least a qualitative measurement of changes that occur during storage. Under conditions used in these experiments, only a small amount of material was viewed by the instrument and, as a consequence, there was considerable variation between readings. When several readings were averaged, fairly consistent values were obtained.

Reflectance measurements appear to be promising as a research tool to determine influence of storage, blanching, and pre-processing conditions on color of vegetables. Certain problems are encountered here, however. The instrument does not recognize variability between color of different pieces of vegetables but merely gives an overall color measurement. It has also been noticed in this work that variation in roughness or in the amount of moisture on the surface of the vegetable has a marked effect on the values obtained.

SUMMARY

Commercial lots of frozen peas, green beans, and cauliflower obtained from the principal producing areas were held at temperatures ranging from 0° to 30° F. Various tests for changes in certain constituents were applied to determine the relationship of the results to time and temperature. Reduced ascorbic acid was found to be a reliable index of previous storage history but it has limited value unless the original content of samples is known. A summation of reduced ascorbic acid and its oxidation products (dehydroascorbic and diketogulonic acids) is more useful than reduced ascorbic acid alone, particularly for green beans. Accumulation of diketogulonic acid as storage progresses seems to parallel adverse changes reasonably well, but more data are needed before more definite conclusions can be drawn. Conversion of chlorophyll to pheophytin was found to be well correlated with temperature experience but is complicated by wide variations in amounts of chlorophyll lost during processing. Measurements of hue and saturation of color of peas and green beans by the Hunter Color Difference Meter show progressive changes related to temperature experience. In spite of rather large experimental error in the method used and initial variations in color between samples, this procedure offers considerable promise.

Acknowledgment

The suggestions and interpretation of reflectance data made by H. C. Lukens and M. M. Boggs are gratefully acknowledged.

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Effect of Reducing Agents on Cured Meat Color[a, b, c]

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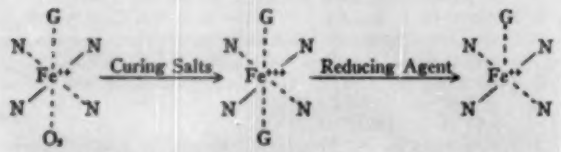
(Manuscript received June 23, 1956)

SINCE ANCIENT TIMES salt has been used to keep meat from spoiling. Salt causes the loss of water and aids in the preservation of meat. However, it has an undesirable effect on meat since it speeds up the chemical change whereby the red meat pigments, myoglobin and oxymyoglobin, are oxidized to the brown pigment metmyoglobin. In early times nitrate was added to meat. The nitrate was reduced by bacterial action to nitrite, which was then further reduced to nitric oxide. Myoglobin, which can combine with oxygen or carbon monoxide, in a similar way combines with nitric oxide to form red colored nitric oxide myoglobin *(8)*.

In modern times curing salts containing nitrite, when mixed with meat and heated, bring about the formation of nitric oxide myohemochromogen [d] producing the so-called "cured meat color" *(6)*. This compound, as mentioned above, is a ferrous heme derivative similar to oxymyoglobin except that oxygen is replaced by nitric oxide *(10)* and the protein is usually denatured.



Oxymyoglobin (Red) — Metmyoglobin (Brown) — Nitric Oxide Myoglobin (Red)

In this reaction, it is known that the addition of nitrite to oxymyoglobin causes the formation of brown metmyoglobin. Heat treatment following the addition of nitrite accelerates the conversion of brown metmyoglobin to pink, denatured nitric oxide myoglobin. The formation of this "cured meat" pigment requires a reducing agent to reduce nitrite to nitric oxide, and metmyoglobin to myoglobin since no reaction takes place between metmyoglobin and nitric oxide. This reducing agent may be an integral part of meat; sulfhydryl groups released by heat denaturation have been shown to function in this way *(13)*. However, if reducing agents are supplied from other sources, red undenatured nitric oxide myoglobin forms without heat treatment *(12)*.

Ascorbic acid, a strong reducing agent, catalyzes the formation of nitric oxid myoglobin with or without the use of heat *(14, 15)*. In the present study, the ability of several other reducing agents to produce and protect the pigment of cured meat was investigated. Since ascorbic acid is a reducing agent of the enediol type, two other compounds of this type were compared, i.e., dihydroxy fumaric acid, a trans enediol, and dihydroxy maleic acid, the cis isomer. Reducing agents of the sulfhydryl type, cysteine and glutathione, were also tested. All compounds were tested for their ability (a) to catalyze the formation of the cured meat pigment in hemoglobin solutions, (b) to protect cured meat surfaces from fading, and (c) to regenerate the pigment in light faded cured meat.

MATERIALS

All compounds tested in this series of experiments were C.P. grade.

Previous work has shown that a 0.1% (5.7×10^{-3} M) concentration of ascorbic acid was sufficient to catalyze the production of nitric oxide hemoglobin *(15)*. All compounds were adjusted to pH 5.7 and made up to a concentration of 5.7×10^{-3}M in order to compare the test compounds with the established ability of ascorbic acid in producing and protecting the pigment of cured meat. The percentage concentration of each solution was as follows: ascorbic acid 0.1%, dihydroxy fumaric acid and dihydroxy maleic acid 0.08%, cysteine 0.06% and glutathione 0.27%.

METHODS AND RESULTS

Experiment I. *The production of nitric oxide hemoglobin by use of various reducing compounds.* Hemoglobin solutions were prepared from washed red cells from defibrinated hog blood. The cells were laked with 4 parts of distilled water and stored in a freezer at −20° C. At the time of use the pH was adjusted to 5.7 with dilute HCl, and the stroma material filtered off before dilution to a final concentration of 1 part red cells to 40 parts of water. This concentration was found to be suitable for colorimetric readings.

The final solutions also contained 0.02M phosphate buffer, pH 5.7, and 0.1% sodium nitrite in addition to the reducing agent specified. Since all readings were to be made in an Evelyn colorimeter, the solutions were stored directly in Evelyn colorimeter tubes. All readings were made using a filter transmitting at 635 mμ. An increase in transmission indicates conversion of methemoglobin (formed immediately upon contact with nitrite) to nitric oxide hemoglobin *(15)*. For the positive control sufficient hydrosulfite was added to a buffered hemoglobin solution containing 0.1% nitrite to give immediate conversion to nitric oxide hemoglobin. The negative control was obtained by addition of 0.1 ml. of 20% ferricyanide to a buffered hemoglobin solution containing 0.1% nitrite. This treatment maintained all of the pigment in the form of metmyoglobin. The positive and negative controls were stable during the course of the experiment.

The comparative ability of the various compounds to catalyze the production of nitric oxide hemoglobin was followed by transmission readings and reported in Table I.

Experiment II. *Regeneration of the pigment of cured meat after light fading.* It has been shown that faded meats which contained sulfhydryl groups could be restored to the original pink of the "cured meat" pigment by dips into nitrite solutions.

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[a] Presented before the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 12, 1956.

[b] This paper is derived from a dissertation submitted by George G. Kelley to the Graduate Council, Florida State University, in partial fulfillment of the requirements for the degree of Doctor of Philosophy. The complete report is in the Florida State University Library, Tallahassee.

[c] Work financed in part by a grant from Swift and Company to the Florida State University.

[d] In heat treated cured meat the globin is denatured and the pigment produced is called nitric oxide myohemochromogen. However, for clarity the term myoglobin will be used throughout this publication.

Meats which did not contain available sulfhydryl groups could not be restored to their original pink color *(13)*. This experiment was designed to note and compare the effect of various reducing agents on meat containing residual nitrite, but lacking available sulfhydryl groups.

A test for residual nitrite has been described by Erdman and Watts *(3)*. 0.2 ml. of a sulfanilic acid-acetic acid solution, (10 g. of sulfanilic acid in 1000 ml. of 30% acetic acid) and 0.2 ml. of α-naphthylamine-acetic acid *(4)* (0.03 g. of α-naphthylamine in 70 ml. of water added to 30 ml. of glacial acetic acid) is dropped on filter paper (Whatman No. 54 which is nitrite negative according to this test) and the filter paper is pressed onto the surface of the meat. A pink color on the filter paper denotes residual nitrite. The depth of the color is proportional to the amount of nitrite.

To test for sulfhydryl groups, a drop of concentrated ammonium hydroxide, followed by a drop of 5% nitroprusside was added to the surface of the meat. A deep rose color developed in the presence of free sulfhydryl groups *(2)*. Those meats which did not develop any trace of the rose color were judged suitable for this experiment.

The positive control, arbitrarily designated 10, consisted of a freshly cut, nonfaded surface of the meat in question. The negative standard, designated zero, was obtained by soaking a piece of the same meat in a 1% solution of potassium ferricyanide for 5 minutes, then rinsing off excess ferricyanide with distilled water. The brown color obtained after this treatment was assumed to represent complete oxidation of the heme iron to yield ferri hemochromogen.

TABLE 1

The effects of reducing agents on formation of nitric acid hemoglobin at varying temperatures

Compound	Temperature 0° C.	Peak transmission at 635 mμ	Time to reach peak	Duration of reading
Positive Control	4°	83	1 min.	7 days
	20°	83	1 min.	5 days
	40°	83	1 min.	2 days
Negative Control	4°	24	10 min.	Indefinite
	20°	24	10 min.	Indefinite
	40°	24	10 min.	Indefinite
Ascorbate 5.7×10^{-2} M	4°	80	90 min.	7 days
	20°	83	50 min.	5 days
	40°	83	30 min.	2 days
Dihydroxy fumarate 5.7×10^{-2} M	4°	67	180 min.	1 day
	20°	65	80 min.	1 day
	40°	67	45 min.	1 day
Dihydroxy maleate 5.7×10^{-2} M	4°	24[1]	.....	.....
	20°	24	.....	.....
	40°	24	.....	.....
Cysteine 5.7×10^{-2} M	4°	85	120 min.	4½ days
	20°	85	55 min.	3¼ days
	40°	83	25 min.	2 days
Glutathione 5.7×10^{-2} M	4°	85	85 min.	6½ days
	20°	85	55 min.	5 days
	40°	87	45 min.	2½ days

[1] Precipitate formed within 1 hour.

Cured meat (bologna) which gave a nitrite positive test and sulfhydryl negative test, was sliced and placed under a layer of polyethylene to prevent loss of moisture. The sliced meat was then placed under a lamp and subjected to 125 foot candles of light until it faded to a grey-brown color comparable to that of the negative control.

The meat was immediately dipped for 30 seconds in a solution (5.7×10^{-2}M, adjusted to pH 5.7) of one of the reducing agents listed above. This concentration is ten times greater than used before since these solutions were used for a quick dip.

After dipping, the meat slices were covered with polyethylene and stored in the dark in the refrigerator. Color regeneration was observed visually by comparisons with the appropriate controls. The results are shown in Figure 1.

Experiment III. *Protection of "cured meat" pigment from fading.* This experiment is similar to Experiment II. Instead of regenerating the "cured meat" color, an attempt was made to protect the pigment from fading by addition of the various reducing agents. The same methods and concentrations of the reducing agents were followed in this experiment as were followed in Experiment II.

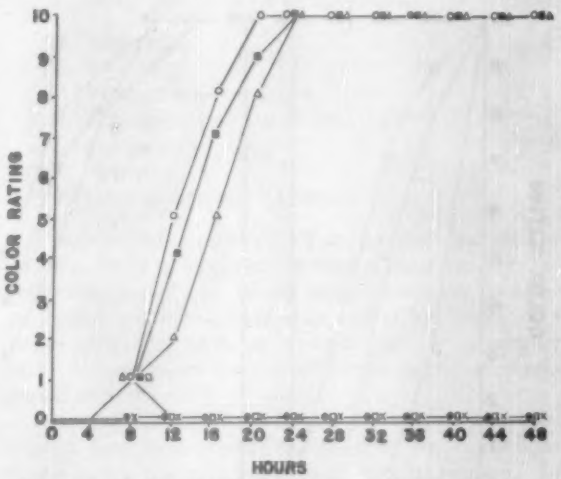

Figure 1. Action of reducing agents in the regeneration of the color of faded cured meat.

Legend

- △ Ascorbic acid
- ○ Cysteine
- ■ Glutathione
- ● Dihydroxymaleic acid
- □ Dihydroxyfumaric acid
- × Control

Freshly sliced bologna with good red color, positive nitrite test, and negative nitroprusside test was dipped for 30 seconds in one of the reducing agents. The slices were covered with polyethylene and placed under 50 foot candles of light. Color fading was observed visually by comparisons with the appropriate controls. The results appear in Figure 2.

DISCUSSION

Three of the reducing agents tested, ascorbic acid, cysteine and glutathione, were capable of producing the red "cured meat" pigment in the presence of nitrite. At the same molar concentrations, the three compounds were about equally effective under all conditions of experimentation, i.e., in (a) the formation of nitric oxide hemoglobin in solution, (b) the regeneration of nitric oxide myoglobin on light faded meat surfaces, and (c) the protection of fresh surfaces from fading.

The mechanism involved in the production of the pigment of cured meat is still obscure. It is possible that an intermediate is involved. It has been demonstrated *(9)* that nitric oxide combined not only with reduced hemoglobin to give nitric oxide hemoglobin, but also with methemoglobin to give an unstable nitric oxide methemoglobin wihch undergoes a slow autoreduction to nitric oxide hemoglobin. In addition Barnard *(1)* and Watts *(11)* have presented evidence for

the formation of a methemoglobin nitrite to explain changes from a brown to a red color in solutions containing a high ratio of nitrite to hemoglobin. Thus the reducing agents may act on such an intermediate to give the cured meat pigment or they may act directly on nitrite and metmyoglobin to give nitric oxide and myoglobin, which then combine. There is evidence for the the reduction of both nitrite *(7)* and metmyoglobin *(5)* by ascorbic acid.

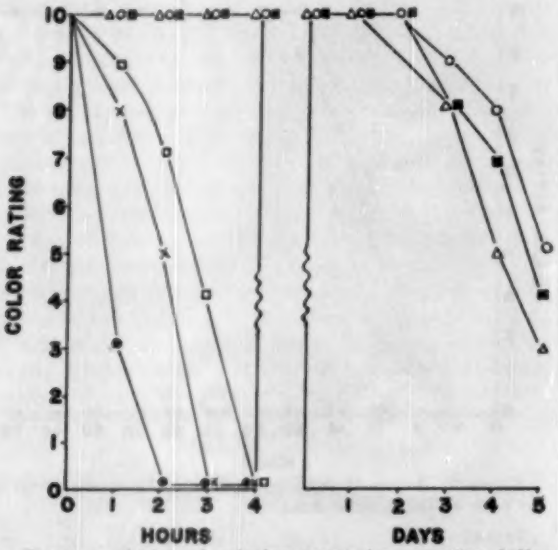

Figure 2. Action of reducing agents in preventing fading of "cured meat" color.

Legend

- △ Ascorbic acid
- ○ Cysteine
- ■ Glutathione
- ● Dihydroxymaleic acid
- □ Dihydroxyfumaric acid
- × Control

Since the three successful reducing agents gave approximately the same results on a molar basis, their effectiveness on a weight basis is inversely proportional to their molecular weights. Thus, cysteine may be considered 2.5 and ascorbic acid 1.7 times as effective as glutathione per unit weight. From the standpoint of possible additives to meat, only ascorbic acid is practical at the present time because of cost. However, the concentration of cysteine in completely denatured myosin is 1.1% or approximately 0.2% in meat. Glutathione comprises about 0.05% of muscle tissue. The sulfhydryl content of the heat denatured meat should therefore be extremely effective in the production of nitric oxide myohemochromogen, and advantageous for its protection after it is formed.

Dihydroxy fumaric acid, a trans enediol, and dihydroxy maleic acid, the cis and most unstable isomer, were not effective in catalyzing the pigment of cured meat in nitrite hemoglobin solutions, nor were they effective in protecting or regenerating the nitric oxide myohemochromogen on the surfaces of sliced cured meat. In all cases studied the action of these two enediol compounds led to products which compared in color to the zero control or in some cases to greenish compounds, suggesting porphyrin ring destruction. Previous work *(16)* has shown that enediols, in the presence of copper ions are oxidized with the production of H_2O_2. It is possible that these two enediols which are quite unstable, may yield H_2O_2 as a primary product. This could account for the greening.

SUMMARY

Three of the reducing agents tested—cysteine, ascorbic acid, and glutathione—were capable of (a) catalyzing the production of nitric oxide hemoglobin (b) regenerating the nitric oxide myoglobin on the surfaces of light faded cured meat when residual nitrite is present, and (c) protecting the surfaces of cured meat from fading by light. The effectiveness of the three compounds on a weight basis is inversely proportional to their molecular weight.

Dihydroxy fumaric acid and dihydroxy maleic acid are too unstable for use and lead to the formation of green, grey-brown products.

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A Practical Look at Automation For the Food Plant[a]

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THE TERM "AUTOMATION," as generally accepted today, began to be used with some degree of frequency about two or three years ago. It was immediately snapped up by economists, industrialists, and labor leaders and basically redefined to suit their own concepts or needs. The fact that each viewed automation from the perspective of his own experience has created, in my opinion, some distortion in the overall concept. How much of my own outlook toward automation is colored by my engineering background is a matter for conjecture.

Myriads of words have been used in describing the wonders of automation. We all have gone to great lengths at times to determine the potential effects of automation in our plants; in this approach we have clouded our views by thinking of automation in its more complicated forms. I shall not concern myself in this paper with the complexities of a mythical push-button plant—a plant with a completely automated tie-in from supply to delivery points, fabulous control rooms, and fully automatic office procedures. Neither will I leave you with the impression that such a plant is totally visionary. Rather, I shall attempt to deal with an interpretation of automation in a practical vein—automation as a direct means to simple solutions of the specific problems so common in our plants today.

Effective quality control and economies of operation may be achieved by primary yet functional applications of automatic control. In our planning to achieve the greatest benefits of automatic control, we must not limit ourselves to orthodox thinking. It can be proved that the greatest economies are usually obtained when some radically different method of doing a job is devised. These methods have been made feasible by the many recent advances in technology and equipment design. Normally, in going from a manual to an automatic operation, we find that it is much simpler to do the job the new way. One of the outstanding characteristics of automation is the fact that many unexpected benefits frequently result.

We must recognize the fact that, as far as quality control is concerned, we are faced with many problem situations when dealing with many of our food products. Properties of foods are not sharply defined but vary over a rather large range. Inspection and quality ratings under these conditions have been usually left to human judgment, based on the experience of the feel, look, the texture of the product. Experimentation can help to make our judgment more perfect; techniques developed in our laboratories and quantitative measures can frequently be shown to be related to the quality required.

We should also remember that there probably is more rigid tradition in the food industry than in other industries. This is due in great part to the dependence on customer usage and acceptance. We find that we may be bucking precedent, custom—and, in rare instances, law—in our attempts to make changes in methods.

A typical pattern. It is generally conceded that to obtain automatic control or automation in most processes the following pattern must be developed:

1. Material in
2. Process
3. Product out
4. Sensing instrument or feed back
5. Computor
6. Actuator
7. Back to Step 1—"Material in"

Measurements are made on the product leaving the process. If there has been a deviation from the standard conditions as set up, the automatic controller or computer determines the corrective action by means of a power actuator, such as a motorized or pneumatic valve, to restore the output to normal in the minimum period of time.

In a general sense we interpret automation as a continuous automatic production based on the continuous flow concept instead of the batch system concept. In many phases of food processing, however, a continuous effect can be obtained through the use of modified batch systems. To what degree we should automate must be determined as much by economic as by technological considerations.

Automation does not by necessity become of paramount importance for large production problems only. Effectively engineered, practically applied, and combined with equipment of proper design, automation then may no longer be classified as a luxury item, but rather as a means to attain economically a repetitive quality of product to meet specific conditions as set up by improved techniques developed in the research divisions and in our laboratories. This is particularly true in the case of the small plant operator. There can be no doubt that many companies using self-regulating equipment or automatic control will obtain a marketing edge over their competitors. Others must follow suit or fall by the wayside. The only processes that would not benefit from some sort of automation would be, in my opinion, those having yields of 100% or better and a zero labor cost.

In the same practical vein, we can say that automation necessarily includes all those things that increase productivity, reduce cost and give us better quality control. This must include, among other things, process equipment, materials handling, building layout, automatic machinery, automatic controls, and instrumentation. At times we lose sight of the importance of suitably designed equipment. *We are prone to accept equipment that is readily available at so-called standard pieces, rather than to include in our analysis of the process at hand the feasibility of applying equipment of*

[a] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 12, 1956.

proper design. This is especially true in the incorporation of the sanitary type of construction. By necessity sanitary design is also the more simple design. Although this may be the more expensive approach, I feel that such is the exception rather than the rule. The more functional the design and use of our process equipment becomes, the more simple the solution to the overall problem at hand. Each phase must be examined in a similar way to determine what is available or can be developed, and each must be integrated to fit the overall objective if we are to realize success in an automated set-up.

There is a simple and practical approach to automation as applied to processing. This basic approach has proved applicable not only to simple phases of corrective automatic control or the development of food processing equipment but also to the creation of a complete processing set-up. This approach normally would fall into the following steps:

1. The objective
2. A review of known and anticipated conditions
3. The scheme
4. The design

The ultimate test. The ultimate test of this approach would be to determine whether or not these integrated steps would inversely satisfy the original objective. Not only does this formula lead to the creation of a solution which satisfies the original objective, but it is also definitely instrumental in helping to create a system that accomplishes this objective in the most practical manner. What is frequently referred to as a "problem" is often an opportunity to create a better functioning or a more practical system. To view automation in terms of a problem alone is to approach the ultimate solution negatively. True, automation cannot just be talked into being, or purchased into being out of a catalog. It can only be attained through the whole hearted endorsement of far sighted management.

In my own company we are constantly on the alert for fresh and original ideas. In getting the originality we seek more quickly, we take advantage of the present trend to "team engineering." As our use of automated systems increases, there will be a diminishing need for engineering specialization and a greater need for the general knowledge and experience resulting from the development of the team engineering concept.

The Relation of the Calcium Content of Potato Tubers to the Quality of Potato Chips[a, b]

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(Manuscript received August 1, 1956)

VARIETAL DIFFERENCES ARE KNOWN to be responsible for much of the variation in the quality of potato chips. Specific physical and chemical constituents which bring about these differences have been under investigation by personnel at the Ohio Experiment Station since 1953. A study of the factors influencing the color of potato chips has been published *(5)*. Another, on the role of specific amino acids and specific reducing sugars, has been prepared. The present paper includes the relation of calcium, as found in the tubers of 32 varieties of potatoes, to the fat absorption and color of chips made therefrom. It also reports data on the effects of moisture levels, planting dates, and nitrogen levels on the calcium contents of potato tubers.

Use of calcium for firming canned tomatoes is now rather general *(7, 8, 9)*. Calcium salts are also employed to a limited extent for firming other tissues *(7, 13)*. Pyke and Johnson *(11)* found that sloughing of boiled potatoes could be satisfactorily controlled by soluble calcium salts. The U. S. Food and Drug Administration has approved (*Federal Register*, May 10, 1949) the use of several calcium salts for canned potatoes. The role of pectin and its calcium compound has been reviewed by Kertesz *(7)*. According to this review, ordinary gels are formed largely through hydrogen bonding (i.e., between the H of high ester anhydrogalacturonic acid chains and the OH of the sugars) whereas gels from low ester pectins are formed largely through ionic bonding by the aid of calcium and other multivalent ions. Dastur and Agnihotri *(4)* reported a progressive increase of total pectic substances during growth of the potato tuber but during storage the soluble pectin increased and total pectin decreased. Bettelheim and Sterling *(1, 2, 3)* found the greatest amount of calcium in the HCl-soluble fraction and the next greatest in the Calgon-soluble fraction of potato tubers. Cooking caused a decrease in "intrinsic viscosity," in methoxyl content of calcium pectinate, and calcium content of calcium pectinate. They found a high correlation between starch content of cooked potatoes and organoleptic scores, but no correlation between the chemical nature of the starch and potato texture.

EXPERIMENTAL METHODS

Six varieties of potatoes which grew at 3 locations—Wooster, West Lafayette, and Coshocton, Ohio (a total of 15 lots)—were included in the 1954 tests. Thirty-two varieties which grow at Wooster, were included in the 1955 tests. Potatoes at Wooster and Coshocton grew without benefit of supplemental irrigation. Those at West Lafayette received supplemental irrigation—i.e., were irrigated approximately 7 days after each rain or irrigation. The Russet Rural variety was planted at Wooster, Ohio, in 1954 on three dates—May 6, May 20, and June 10. An additional experiment on the effect of nitrogen was conducted at

[a] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 13, 1956.

[b] Journal Article No. 55-56, Ohio Agricultural Experiment Station, Project: Hatch 122-3.

Wooster on the Katahdin variety. Samples from Coshocton and West Lafayette were from non-replicated lots. Those from Wooster were composites of 4 replications. Thus, 15 lots were included in the 1954 tests and 32 in the 1955 tests.

Peeling. Twelve to 15 potatoes were chosen at random from each experimental lot and peeled for 3 minutes in an abrasive peeler. A ⅜-inch cork borer was forced through the center of each potato from stem to terminal bud end. The hole thus made was used in supporting the cut pieces during frying.

Slicing. Stem-end and apical-end thirds of each peeled potato were discarded. Center third of each potato was then sliced in a rotary-type, hand-operated slicer. Slicer blade was previously set so that slices of approximately 1/16-inch in thickness were produced.

Washing. All slices from each sample were then washed in cold, running, softened tap water to remove loose starch and then blotted dry with a clean, dry towel.

Storage. All lots were held in common storage at Wooster until early (Aug. 25) as well as late (Oct. 5) maturing lots were dug. Temperature of this storage varied between 68° and 48° F. Tubers were trucked to Columbus on November 5. Since immediate frying tests indicated a slight sugar accumulation, the tubers were held for 12 days at room temperature (70°-75° F.) for reconditioning prior to frying. After this period frying of all lots was completed within 20 days.

Frying. Quadruplicate samples of 100 g. each were chosen at random from each lot immediately prior to frying. Each sample was placed separately in wire baskets, after which a stainless steel wire was passed through the holes in the slices in order to hold them horizontally in the oil during frying.

A thermostatically-controlled Hotpoint Inc. No. 101 HK 3 Ser. 993969 197-219 Volt frier was used for frying. Two baskets containing the 100-g. samples were dropped simultaneously into the fryer, which contained 15 pounds of fat (Primex), when the thermometer indicated a temperature of 375° F. At this point the electric current was turned off. A stop-watch was used to time the frying period.

End point was determined as described by Sweetman *(11)* and Wright *(14)*, i.e., when the chips became opaque and emitted smaller bubbles. At this point the samples were removed, simultaneously, from the frying medium and the frying time and final temperature recorded. The baskets were then attached to a support provided with the fryer, and the excess oil was allowed to drain. Chips were weighed before placing in .0015 in. polyethylene See-Safe bags and sealed with rubber bands. The chips were held in these bags until evaluated for color and fat content. All evaluations were made within 30 days of the time the chips were fried.

Color evaluation. All potato chip samples were evaluated by 10 judges. Each judge was asked to evaluate numerically the relative color of each sample. The Color Reference Standard made by F. J. Coughlin, Procter and Gamble Co., was employed. The samples of chips were compared with the reference chart and evaluated from 1 to 10. Number 1 represented a very pale color, and Number 10 represented a dark brown color.

Chemical evaluation. Fat was determined by the official A.O.A.C. method, employing a Bailey-Walker continuous extraction apparatus. Two of the 4 potato chip samples, after color values had been determined, were ground finely in an Osterizer for several minutes (60 g. in each sample), until the chips had the appearance of corn meal. Duplicate 2-g. samples of the homogeneous mixture were extracted with petroleum ether for 8-12 hours.

Total solids. Four samples of 100 g. each were selected at random from sliced potatoes, described above, and tested for total solids according to the method outlined by Bettelheim and Sterling *(2)*.

Pectic substances. Preparation of "water-soluble" extract. In 1954, water-soluble extracts, made according to the method used by Bettelheim and Sterling *(3)* were found to contain but negligible contents of calcium. This procedure was therefore omitted in 1955.

"Calgon" and "acid-soluble" extracts. The procedure described by Bettelheim and Sterling *(3)* was also used for extraction of the "Calgon" and "acid-soluble" fractions. The Beckman DU Flame Spectrophotometer was employed for the calcium determinations. Flame analyses were carried out directly upon the "Calgon" and "acid" extracts, using the following wave-length and slit setting:

Wave-length millimicrons	Slit mm.	Rel. int. range	p.p.m. range
422	0.01	6-100	0-300

RESULTS

Total solids. Results of the total solids determinations of 15 samples from West Lafayette, Coshocton and Wooster, are shown in Table 1. It can be seen from this table that Russet Rural from Coshocton (non-irrigated plot) had the highest total solids content (25.1%), and Katahdin from West Lafayette (irrigated plot), Michigan 1363, and Red Pontiac, had low total solids, with ranges from 17.1-18.8%. In general, total solids content as shown in Table 1 agree with data presented elsewhere *(10)* which showed that total solids or specific gravity of potato tubers decreased with supplemental irrigation and increased with later data of planting and that there was a significant difference in the total solids of some varieties.

TABLE 1

Calcium and total solid content of potato tubers; color values and percent fat absorption of chips from potato variety samples from West Lafayette, Coshocton and Wooster, Ohio—1954

Variety	Calcium (mg./100 g.) potatoes, dry weight basis. Ave. 2 det.			Total solids percent Ave. 4 det.	Color values* Ave. 4 det.	Fat absorption percent Ave. 2 det.
	Calgon extract	Acid extract	Total			
Russet Rural[1]	25	34	59	25.1	6.0	40.6
Russet Rural[2]	22	38	60	23.3	6.0	39.3
Russet Rural[3]	13	30	43	20.5	6.5	43.1
Russet Rural[4]	15	25	40	23.4	7.0	43.7
Russet Rural[5]	18	21	39	23.4	7.0	44.7
Katahdin[1]	16	20	36	23.5	6.5	41.7
Katahdin[2]	11	19	30	17.1	7.0	42.9
Katahdin[3]	15	24	39	21.5	7.0	45.0
Katahdin[5]	13	15	28	19.0	6.0	42.3
Katahdin[6]	13	17	30	21.0	7.0	43.6
Katahdin[7]	16	22	38	21.4	5.0	41.8
Michigan 1363[2]	2	12	14	17.5	9.0	50.1
Redkote[2]	12	20	32	20.2	5.0	42.0
Red Pontiac[2]	2	8	10	18.8	9.0	49.4
Sebago[2]	9	21	30	24.0	7.0	46.0

[1] Not irrigated from Coshocton.
[2] Irrigated from West Lafayette.
[3] Not irrigated from Wooster, planted May 6.
[4] Not irrigated from Wooster, planted May 20.
[5] Not irrigated from Wooster, planted June 10.
[6] Not irrigated 160 #N/acre.
[7] Not irrigated, no nitrogen.
* Color values based on color reference standard by F. J. Coughlin, Procter and Gamble Co.
(1 = *light;* 10 = *dark*).

Color values. Color values of chips as shown in Table 1, column 6, indicate that Redkote Russet Rural and Katahdin (no nitrogen plot) yielded light-colored chips, and Michigan 1363 and Red Pontiac yielded dark colored chips.

Fat absorption. The percent of fat absorption shown in Table 1 indicates that the chips made from Red Pontiac and Michigan 1363 had a high fat absorption (from 49.4-50.1%), and the chips made from Russet Rural (irrigated and non-irrigated field) had a low fat absorption (from 39.3-40.6%).

Calcium content. Results of the calcium determinations of samples from "Calgon extract" are shown in Table 1. Russet Rural had the highest calcium content (25 mgs. per 100 g. dry weight), and Red Pontiac and Michigan 1363 had very low calcium content (2 mgs. per 100 g. dry weight).

Results of calcium determinations of samples by acid extraction are shown in Table 1. Russet Rural from irrigated and non-irrigated fields had high calcium content (34-38 mgs. per 100 g. dry weight), and Red Pontiac and Michigan 1363 had low calcium content (8 and 12 mgs. per 100 g. dry weight).

It is interesting that there were great differences in total calcium content among the varieties, 10-60 mgs. per 100 g., but less among the lots of the same variety (Table 1). For instance, Russet Rural (from irrigated and non-irrigated fields) had high total calcium content (59-60 mgs. per 100 g. dry weight), while Red Pontiac and Michigan 1363 had low calcium content (10 and 14 mgs. per 100 g. dry weight). Total calcium content of the 5 samples from Russet Rural range from 39-60 mgs. per 100 g. dry weight, and the lots from Katahdin range from 28 to 39 mgs. per 100 g. dry weight.

Based on these results, it was deemed advisable to make a further investigation of the total calcium content of potato varieties in 1955. The results of these tests, including 32 varieties, are shown in Tables 3 and 4. The data in these tables confirm those presented in Tables 1 and 2.

TABLE 2

Coefficient of correlation between total calcium content and total solids of potato tubers, color values and percent fat absorption of chips (data based on Table 1)

Gross coefficient	Coefficient of determination percent value	First order partial coefficient	Second order partial coefficient	Multiple coefficient	Determination percent value
$r_{c.1} = 0.64$	42.32	$r_{c1.2} = 0.58$ $r_{c1.3} = 0.59$	$r_{c1.23} = 0.57$		
$r_{c.2} = -0.69$	48.02	$r_{c2.1} = -0.64$ $r_{c2.3} = -0.29$	$r_{c2.13} = -0.35$	$R_{c.123} = 0.93$	86.30
$r_{c.3} = -0.87$	76.10	$r_{c3.1} = -0.85$ $r_{c3.2} = -0.75$	$r_{c3.12} = -0.75$		

Legend:
c—Calcium.
1—Total solids.
2—Color value.
3—Percent fat absorption.

The coefficient of correlation between total calcium content of potato tubers (15 lots) and total solids (0.64) is shown in Table 2 (data based on Table 1). Coefficients of correlation are also included between total calcium content and the color values (—0.69) and the percent of fat absorption (—0.87) of chips made from these lots. It can be seen from this table that a highly significant negative gross correlation coefficient of —0.87 is found between total calcium of the tubers and percent fat absorption of chips. This indicates that the higher the calcium content of tubers the lower the fat absorption of chips made therefrom. The percent value of determination of this comparison is 76.10, which indicates that 76.10% of the variability of fat absorption of chips can be explained by variation in calcium content of the tubers. A significant negative gross correlation coefficient of —0.69 was also noted between

TABLE 3

Calcium and total solid content of potato tubers; color values and percent fat absorption of chips from potato variety samples from Wooster, Ohio, planted May 6, 1955

Variety	Calcium (mg./100 g.) potatoes, dry weight basis. Ave. 2 det.			Total solids percent Ave. 4 det.	Color values* Ave. 4 det.	Fat absorption percent Ave. 2 det.
	Calgon extract	Acid extract	Total			
Russet Rural	25	37	62	17.9	3.5	43.0
Red McClure[1]	23	37	60	20.9	5.0	36.4
De Sota[1]	27	31	58	19.6	6.1	42.9
Russet Burbank	22	35	57	20.0	4.2	39.8
Erdgold[1]	24	31	55	20.2	4.0	38.6
Pawnee	21	31	52	16.8	5.8	42.2
Progress[1]	23	28	51	19.4	6.8	41.4
Mark Redwitzer[1]	20	30	50	20.3	4.5	37.6
White Rose[1]	20	28	48	21.1	4.8	39.2
Saco[1]	18	29	47	19.9	4.5	39.5
Ontario	20	26	46	19.2	5.1	42.6
Jacobi[1]	19	27	46	20.8	7.1	38.8
Canoga	17	25	42	18.4	6.4	40.8
Cobbler	20	21	41	18.5	6.0	45.1
Pungo	22	18	40	16.7	5.8	43.8
OB-638-16	17	23	40	15.5	6.8	44.4
Rushmore[1]	16	24	40	16.2	6.4	45.5
White Cloud	15	23	38	16.5	4.8	45.8
Waseca	18	19	37	15.5	6.8	47.0
Louisiana 92-23[1]	16	20	36	15.4	5.0	46.1
Alpha[1]	14	22	36	16.7	5.1	47.4
Kennebec	13	22	35	14.6	5.1	45.0
Teton	10	24	34	15.2	6.6	46.8
Chippewa	13	20	33	17.1	3.7	45.5
Redkote	9	23	32	17.3	4.1	46.0
Sebago	13	17	30	17.1	4.9	45.0
Redburt	8	22	30	14.0	8.1	47.9
La Soda	11	19	30	13.8	6.6	45.1
Cherokee	10	20	30	16.1	4.1	45.8
Katahdin	13	17	30	15.3	4.2	46.0
Red Pontiac	8	14	22	14.1	8.0	52.2
Michigan 1363	7	13	20	13.8	9.2	47.9

[1] Planted June 3.
* Color values based on color reference standard by F. J. Coughlin, Procter and Gamble Co. (1 = *light*; 10 = *dark*).

total calcium of the tubers and the color value of chips. However, data secured in 1955 (Table 4) gave a correlation of —0.40 which is significant only at the 5% level. This tends to indicate that the higher the calcium content of tubers the lighter the color of chips made therefrom. The percent value of determination of this comparison is 48.02. The significant positive gross correlation coefficient of 0.64 (Table 2) between the total calcium and the total solids of the tubers indicates a direct relationship between these two factors. The per-

TABLE 4

Coefficient of correlation between total calcium content and total solids of potato tubers, color values and percentages of fat absorption of chips (data based on Table 3) 32 varieties—1955

Gross coefficient	Coefficient of determination percent value	First order partial coefficient	Second order partial coefficient	Multiple coefficient	Determination percent value
$r_{c.1} = 0.80$	64.00	$r_{c1.2} = 0.76$ $r_{c1.3} = 0.33$	$r_{c1.23} = 0.31$		
$r_{c.2} = -0.40$	16.00	$r_{c2.1} = -0.09$ $r_{c2.3} = -0.16$	$r_{c2.13} = -0.10$	$R_{c.123} = 0.82$	67.24
$r_{c.3} = -0.81$	65.61	$r_{c3.1} = -0.77$ $r_{c3.2} = -0.38$	$r_{c3.12} = -0.37$		

Legend:
c—Calcium.
1—Total solids.
2—Color value.
3—Percent fat absorption.

cent value of determination of this comparison is 42.32. Corresponding correlation for the 1955 data is 0.80 (Table 4).

In the 1954 data negative first order partial correlation coefficients of —0.85 and —0.75 were found between the calcium content of the tubers and the percent fat absorption of chips after the elimination of the effects of total solids of the tubers and the color values of chips, respectively. The 1955 data (Table 4) yielded corresponding correlations of —0.77 and —0.38. The second order partial correlation coefficient of —0.75 as shown in Table 2 represents the net effect of calcium after the removal of both the other contributing factors, i.e., total solids and color values of chips. There was little difference in value between the gross and partial correlation coefficients (first and second). Therefore, a persistent relationship is indicated between the calcium content of tubers and fat absorption of chips.

In the 1954 data, negative first order partial correlation coefficients of —0.64 and —0.29 are noted between calcium content of tubers and color values of chips after the elimination of the effects produced by total solids of the tubers and percent fat absorption of chips, respectively. Corresponding values for 1955 were —0.09 and —0.16 respectively (Table 4). Second order partial coefficients of —0.35 in 1954 and —0.10 in 1955 are noted after the removal of the effects of total solids and percent fat absorption of chips. There was a difference in value between the gross and partial correlation coefficients (first and second). Therefore, an interrelationship exists between color value of chips and total solids of the tubers. It is obvious from these data that calcium content of potato tubers had less effect on color of chips than on fat absorption of chips.

Based on these combined factors—total calcium, total solids, color values, and percent fat absorption of chips —the multiple correlation coefficients of 0.93 in 1954 and 0.82 in 1955 were obtained. The values are highly significant and thus indicate that by combining the three factors—total solids of tubers, color values, and percent fat absorption of chips—an index can be secured which may be employed to predict the total calcium content of the potato tubers.

Thus, in this study, high calcium content of potato tubers was more closely correlated with low fat absorption and high total solids than any other factor which was determined.

In calculating partial correlation coefficient, three assumptions have been made: (a) The gross correlation had linear regressions. (b) Each independent variable had an effect on the dependent variable (i.e., total calcium content). (c) These effects were additive.

Gross correlation coefficients showed the relationship between the dependent variable (calcium) and an independent variable which includes the effects of two other independent variables. For example, $r_{c.1} = 0.80$, means that the relation between calcium (c) and total solids (1) which also includes fat absorption and color value, is 0.80, a value which is highly significant for 31 degrees of freedom.

The partial correlation coefficient showed the same relationship after the removal of the effects of one (first order) or two (second order) of the other independent variables. The correlation coefficients for calcium and fat absorption $r_{c.s} = -0.87$, $r_{cs.1} = -0.85$, $r_{cs.2} = -0.75$ and $r_{cs.12} = -0.75$ remained nearly constant which indicated a persistent relationship between calcium and fat absorption regardless of the two other independent variables, total solids and color value. However, if there was a difference, an interrelationship must be assumed between the independent variables.

DISCUSSION

In considering the results reported herein it is important to remember that varieties were chosen which differed widely in attributes known to influence quality. Thus, Red Pontiac was included because it produced tubers of low specific gravity (1.059-1.069). Chips made from these tubers were dark in color and absorbed a large amount of fat; namely, 44.6-50.0%. Likewise, the Russet Rural variety was included because it usually produced tubers which had a high specific gravity (1.071-1.083) and could be fried with reasonable assurance that the chips would be light in color and would not absorb excessive amounts of fat, i.e., 45-52%. These two varieties responded according to the accepted concept that high specific gravity of tubers is commonly associated with light-colored chips that absorb relatively small amounts of fat.

Previous tests made at this Station had indicated that the variety Redkote did not respond in the usual manner. Redkote, in spite of its low specific gravity (1.060-1.073), commonly produced chips light in color and absorbed an average amount of fat (42-46%). On the other hand, Pungo, a variety first tested in 1955, usually produced tubers with a relatively high specific gravity (1.070-1.075), from which only dark-colored chips could be made. However, the chips absorbed relatively small quantities of fat, i.e., 43.8%.

The inclusion of these two varieties thus provided an opportunity to investigate the possibility that the presence or absence of one or more constituents from one or the other of these varieties was responsible for excessive fat absorption or for the production of undesirably dark-colored chips.

Since color and fat absorption of potato chips are commonly used as yardsticks in quality evaluations, this discussion will be confined largely to the effects of the different variables on these two attributes.

Color of potato chips Tables 1 and 3 indicate that total calcium content of potato varieties varied widely and that there was a fairly definite association between high calcium content and light color of chips. However, as indicated under the discussion of fat absorption, the association between high calcium content and good-colored chips is not as pronounced as that between high calcium content and low fat absorption. However, the two-way correlations of —0.69 and —0.40 between calcium content and chip color (significant at the 5% level), do indicate a definite trend. Since color evaluations in these instances were subjective, it is possible that objective measures may give a more pronounced and better relationship between calcium content and color evaluation.

Fat absorption of potato chips. Results of the 1954 test, including 15 individual potato samples of 6 different varieties from 3 locations in Ohio in 1954, are shown in Table 1. Data in this table and Table 3 show that the higher the total calcium content in tubers, the lower the percent fat absorption of chips. This observation suggests many problems requiring future investigation. One of the most important is that of devising an accurate yet speedy test for determining the calcium content of potato tubers. Other problems include the use of calcium salts in chipping operations, to reduce fat absorption.

It is known that fat and oil used for frying may develop free fatty acids through hydrolysis. The fat used for chipping had a free fatty acid of 0.05% or less, and as the fat is used and re-used the free fatty acid may increase to as much as 0.5%. It is postulated that during frying the calcium pectate in the potato tissues may be hydrolyzed, and thus the more active calcium ions may react with free fatty acid of the frying fat to form a calcium soap. Due to its large size, the molecule of the calcium soap might penetrate cell walls more slowly than the smaller sodium or potassium soap. On the other hand, the calcium content of the cell might have other physical characteristics which affect fat absorption during frying. From 1955 data, not shown, it was found that added increments of potassium also depressed calcium absorption of the tubers. Unfortunately, magnesium levels were not a part of this study; hence no data can be presented to indicate the role of magnesium in depressing calcium absorption.

From Table 1 it may be seen that the high nitrogen level slightly depressed calcium absorption of the Katahdin variety. Tubers of lots receiving 160 pounds of nitrogen per acre had a calcium content of 30 mg. per 100 g. dry weight basis, although the tubers from the lot receiving no supplemental nitrogen contained 38 mg. per 100 g. dry weight. If these data are confirmed by subsequent tests, nutritionists and horticulturists will have a basis for further tests with nitrogen levels. Other possibilities include a study of the relationships between the calcium levels of foliage and tubers. If such a relationship exists, it is possible that growers could use foliar analyses in predicting the calcium needs for producing better chipping tubers.

SUMMARY

Results of the total calcium[c] determination of 15 individual potato samples in 1954, and 32 variety samples in 1955, indicated that high total calcium of raw potato tubers was correlated with high total solids, light-colored chips and low fat absorption. Varietal differences were greater than differences produced by any of the factors included in this study. Excellent chipping varieties include Russet Rural, Russet Burbank, Erdgold, Mark Redwitzer, White Rose, and Saco. Very poor chipping varieties include Red Pontiac, Michigan 1363, and Redburt.

[c] In this paper total calcium refers to the sum of "Calgon" and "acid"-soluble calcium.

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Discrimination Techniques in Meat Acceptance Studies[a, b]

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SYSTEMATIC EVALUATION of the consumer's reaction toward consumer products is a relatively new area of research. Yet, by comparison, the general concept of consumer research is a mature science in relation to meat consumer research. A search of the literature reveals that the results of one of the first meat consumer surveys was published in 1926 *(1)* but that no real momentum was evidenced in this area of research until the 1950's.

The lag in the initiation of meat consumer research is not attributable to the lack of need for such research, but to numerous intangible factors. The pooled resources and abilities of many disciplines have greatly accelerated recent achievements in this field.

METHODOLOGIES

Since meat consumer research is new, and since it is beset with peculiar technological considerations, various methodologies have evolved. These methodologies, largely, are adaptations from other product investigations.

Consumer preference for meat involves two distinct preferences: visual and eating. Yet, they must ultimately result in a single expression of preference. The visual appeal of an attractive cut of meat is readily offset if the consumer is disappointed in the eating qualities of the product. Conversely, the potential eating satisfaction of meat is rarely realized if the cut is deficient in visual appeal. Visual and eating preferences, thus are the summation of consumer experiences. Dual visual and eating preference studies of Birmingham, *et al. (4)* and Lasley, Kiehl, and Brady *(6)* report many instances of divergent visual and eating preferences. These results indicate the inability of certain consumers to select meat accurately for desired eating characteristics.

One type of consumer survey is characterized by responses based on the consumer's memory or household records in regard to meat consumption habits, desires, and preferences. Surveys founded on this type of response have been accomplished through personal interviews, telephone interviews, or mail questionnaires. Meat consumption habits were ascertained by Adams *(1)*, Anderson *(3)*, and Peters *(8)* using this interview technique. Reaction toward prepackaged and self-service meats was compiled by Riley *(10)* and Sloop *(12)* through the use of the same technique.

The questionnaire or poll-type survey has the distinct advantage of being simple and economical to conduct. It is most useful as a preliminary test in obtaining an appreciation of the latitude of responses that may be encountered. It may further serve to define consumer habits and to measure consumer knowledge of the product characteristic being considered. The severe limitation of this method is the possibility that a preference may be indicated for a particular variable solely because that is the only possibility with which the consumer may be acquainted. The respondent, who indicates a preference for low quality meat because there is no higher quality meat available where the respondent shops, may actually prefer a different quality if given the opportunity to compare meat of various qualities. Another limitation of poll-type surveys is illustrated in the results of Lasley, Kiehl, and Brady *(6)*, Table 1,

TABLE 1

Subjective color of lean associated with Munsell color readings by cooperators (6)

Color paddle number	Bright red lean
	%
1	23.3
2	13.3
3	26.7
4	6.7
5	6.7
6	3.3
7	10.0
8	.....
9	3.3
No Reply	6.7

who found that nearly one-third of the respondents preferred a bright red color of lean for beef. When these respondents were asked to indicate the Munsel meat color paddle which most nearly corresponded to bright red, the extreme range (1-9) of color paddles was indicated. The subjective indication of bright red color of meat thus had no definite objective reference.

A more stable approach for measuring preference is to permit the respondent to make a side by side comparison of the variable. This technique has been utilized in visual, eating, and sales studies. The most extensive use of this technique has been in conjunction with visual preference studies *(4, 5, 7, 8, 10, 12, 14, 16)* of beef and pork. Birmingham, *et al. (4)* and Lasley, Kiehl, and Brady *(6)*, using the comparison technique, determined both visual and eating preferences. Sales studies by Meyer and Ensminger *(7)* and by Lasley, Kiehl, and Brady *(6)* offered the respondents the opportunity to compare the variables being considered.

Combinations of these visual and eating methods have been utilized at the Missouri Experiment Station in a manner which is not readily apparent by casual observation of the publications *(4, 6, 9, 12)* to date. Three specific types of effort are coordinated in the long range attack on a problem. These are:

1. Laboratory Panel
2. Small Urban Household Panel
3. Metropolitan Panel

[a] Presented before the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 13, 1956.

[b] Journal Series Paper No. 1649, approved by the Director of the Missouri Agricultural Experiment Station.

Laboratory panel. During the early stages of a study, a small laboratory panel is employed to test the products under closely controlled conditions. Laboratory panels concerned with meat studies in the past have been used to rate various sensory attributes and to determine preference evaluation. In contrast, the laboratory panel at Missouri is used almost exclusively for discrimination testing.

The primary means of differentiating categories of beef quality is through a system of grading. Meat grades, particularly beef grades, are widely advertised to connotate a degree of consumer satisfaction. Since the grades are not based on factors designed to parallel consumer desires and since many consumers express dissatisfaction with meat they think to be from the "best" grade, it is desirable to determine the homogeneity of the eating characteristics within and between the existing grades. Discrimination techniques are well adapted to yield this information. Reasonably accurate indications of consumer discrimination can be inferred from small laboratory panels since sensory discrimination is believed to be a much less variable factor than preference.

The methodologies incorporated in the laboratory panel studies are dynamic and are adapted to the problem at hand. However, features of the panel are worthy of mention. Panel composition is derived from two groups—faculty members who are experienced meat tasters and students who have had no previous taste panel experience. Tests of short duration and tests of an exploratory nature are well adapted to the use of faculty tasters. However, an inexperienced student panel is used most commonly for extended tests. Some experience indicates that the student panel has a tendency to exhibit less fatigue during a test continuing for several weeks. Within the limits of practicality the panel membership, whether faculty or student, is constant during the entire period of the test. Products are prepared by standardized methods, served as Trio or AAABB comparisons, and consumed in darkened booths.

The laboratory panel has distinct utility within the limitations described above. Comparison testing, whether by discrimination techniques or other means, suffers from the restriction that it is unrealistic in that it departs from what might be considered normal consumer behavior. It remains, however, an excellent tool for defining grade homogeneity for sensory attributes.

Results from a laboratory panel, because of the severe limitation of numbers and because of the rigidly controlled testing conditions, cannot be construed to represent a general population. Household panels are enlisted to test further the hypothesis under consideration.

Urban household panel. A small urban household panel of 36 to 50 families yields considerable information relative to the results to be expected from a larger and more widely stratified population. The panel is selected using a probability sampling technique; comparison products are delivered by appointment; and visual or eating preferences and ratings are obtained. This small scale effort in every way possible parallels a full-scale metropolitan study. It permits visualization of schedule limitations, product handling requirements, and serves to familiarize supervisory personnel with operating procedures.

Metropolitan household panel. A stratified probability sample composed of 250 to 500 families is enlisted for the metropolitan household panel. A general schedule of socio-economic and related consumer information is gathered. The test product is delivered by appointment two times per week. A schedule accompanies each test package and is picked up at the time of the next delivery. The test packages contain sufficient product for one meal for two adults.

Visual preference surveys have also been conducted in food stores. The stores were selected according to size and general income area. Specially designed insulated boxes were utilized to contain the test display. The product was maintained at meat refrigerator temperatures through convection cooling with dry ice. Color deterioration was prevented by systematic product replacement. Interviewing costs are considerably lower than in household surveys. However, sampling accuracy is also somewhat reduced.

Product definition. The acquisition of representative product and population samples are both of great importance in all consumer studies. Population sampling has received wide attention in the literature whereas definition of the meat utilized in consumer studies often has been extremely vague. The lack of product definition immediately seeds doubt in the mind of the technologists who recognize the extreme limitations imposed on the validity of meat consumer research if the product sample population is not carefully controlled.

Most meat consumer studies have been concerned with graded meat. Graded meat, by definition, embraces a certain latitude of variation in the various factors used as a basis for the grade. In order to make a fair comparison of grades it is necessary to restrict the sample of cuts from carcasses within a particular segment of the grade and within a specified weight range.

In addition to the restrictions of grade and weight, it is equally important to control positional location of the cuts for a fair comparison. Recent publication *(5)* of a consumer preference study of graded beef included photographs of comparison steaks from different grades. The most obvious difference in the steaks was due to positional variation—one steak in the comparison was from the posterior portion of the loin end and the other was from the anterior portion. Positional bias can be avoided by following standardized cutting methods which originate the first cut at a fixed anatomical reference and by pairing comparison cuts according to cutting sequence.

Product handling. Wide variation is encountered in the physical characteristics of meat which requires special consideration for purposes of standardization. All variables which are not being studied must be controlled within very narrow limits. This can only be accomplished through careful attention to details and through standardized procedures. Factors that are of extreme importance in this respect are color of lean, color of fat, amount and distribution of fat, amount of bone, thickness, size, and anatomical origin of cut.

Production and design efficiencies can be realized by freezing the product for certain types of eating preference studies, or for visual preference studies which involve only the distribution or proportion of fat. Freezing permits more time to accumulate product inventories. The product may be frozen as a wholesale cut, tempered to 26° F., cut on the band saw, trimmed, packaged, and returned to the freezer before thawing. Freezing also permits design efficiencies by extending the storage life sufficiently to permit delivery or presentation of "replicate" samples at intervals sufficiently far apart as to reduce materially the probability of memory bias.

Coding. Coding must be designed to render complete information to research personnel and yet not be of informative nature to the respondent. Furthermore, sample identification must be simple and positive if a self administered schedule is utilized. In preference comparisons these requirements are met in our laboratory by using a numeral and alphabet code which indicates the household number, date of delivery, carcass identity, carcass grade, and positional location of the retail cut.

The coding of sample comparisons which are to be cooked and eaten requires the use of a coding device which will withstand the rigors of cooking, which will not impart any flavor, and which will not impair the validity of the comparison in any way. Small aluminum rings clamped around the bone portion of samples are very satisfactory for this purpose. Schedules require the respondent to indicate preference and rating according to the number of rings in the samples.

Defining consumer meat grades. Definition of meat grades in characteristics meaningful and of utility to the consumer is beset with many technological restrictions. Meat grades are presently based primarily on carcass characteristics which are designed to have utility to those in the meat industry. Results indicated in Table 2 were derived from laboratory panel discrimination studies of beef grades and weights. The results are based on over one thousand trio judgments of loin steaks from non-adjacent carcass grade and weight categories. The similarity of successes for between and within comparisons suggests an equal degree of homogeneity within and between these categories. It would thus seem that there is some need to consider definition of meat in terms of eating homogeneity.

TABLE 2

Percentage of successful discriminations by laboratory panels testing loin steaks

Comparison	Successes %
Between Grades	54.12
Within Grades	50.22
Between Weights	45.75
Within Weights	48.87

SUMMARY

Preference studies based on comparison interviewing techniques offer the advantage of insuring acquaintance of the respondent with the latitude of variables involved in a study.

The potential utility of laboratory consumer panels, small urban consumer panels, and metropolitan consumer panels can be utilized most efficiently through a systematic and coordinated approach. The gradual increase in tempo and scale of the different panel phases permits transitionary changes in procedures without appreciable loss of data. Discrimination techniques are well adapted to this pattern of study.

Product standardization techniques are necessary to prevent preference bias due to uncontrolled variables.

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The Determination of Density and Evaluation of Quality in Peas Preserved by Freezing

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QUALITY GRADING OF FROZEN PEAS is based on differences in density between the different grades or maturities. In commercial testing for "tenderness and maturity" *(14)*, thawed peas are skinned and placed in a salt solution; the percent sinkers in 10 seconds largely determines the grade of the sample.

In production lines, the processors use brine flotation to achieve separation of higher from lower grades of peas. Such separations have not always been satisfactory *(1, 3, 13)*.

Both grade testing and brine separation depend on differences in density between the more and the less mature peas. For valid comparisons of density with other methods for measuring pea maturity, we found it necessary to develop and use a more precise and accurate method for determining density in frozen peas than was hitherto described *(4, 6, 7)*.

This paper describes the procedure developed for measuring the density of peas and reviews the various factors that affect the accuracy of the results. Statistical evaluations are presented to show the relation between the density values and taste panel appraisals and the percent of alcohol-insoluble solids of the same peas. The peas were specially selected to represent a wide range of maturities and quality grades (A to D) as well as several varieties, harvests, and growing seasons. An effort was also made to maintain homogeneous samples. For this reason, each sample used for a density measurement was restricted to peas of a narrow sieve-size range.

EXPERIMENTAL

Adopted procedure for density determination. The peas (400-500 g.) were thawed in a covered dish which was immersed in warm water, and brought to 80° F. (26.7° C.) All density determinations were made at this temperature as it was the easiest to maintain under existing conditions.

The density measurement was made as follows: A sample of thawed peas (80-100 g.) at 80° F. (26.7° C.) was weighed to 0.01 gram into a covered, tared stainless steel basket. The basket, submerged in a beaker containing 700 ml. of 8% sucrose solution at 82° F. (27.8° C.), was placed in a desiccator, which was evacuated for 5 minutes at a pressure of 30 mm. of Hg. Boiling of the sucrose solution was avoided. The vacuum was released, the beaker removed, and the weight of the submerged basket and peas taken. Conditions were so arranged that at this point the temperature of the liquid and peas was at 80° ± 0.5° F. (26.7° C.). The weight of the empty basket submerged in the same solution at the same temperature was obtained in each determination. The density of the sugar solution just used was then determined with an accurate and clean hydrometer (which could be read to 0.0002). It should be noted that an error of ±0.0010 in the hydrometer reading, regardless of cause, will result in approximately the same numerical error in the density of peas. With 3 or more samples, the time required is about 15 minutes per sample.

Calculations:

$$d_p = \frac{(d_s)\ (p_a)}{p_a - p_s}$$

where d_p = density of peas at 80° F. (26.7° C.)
d_s = density of liquid = hydrometer reading (80° F.) — 0.0013 [a]
p_a = weight of peas in air
p_s = weight of peas in liquid.

DISCUSSION OF PROCEDURE

Defrosting and sampling of frozen peas. Spray defrosting is not recommended because appreciable losses of solutes due to leaching occur and change the density of spray-defrosted samples *(16)*. Leaching losses are greater with less mature than with more mature peas. Therefore, peas were dry-defrosted, i.e., frozen peas in a covered dish were placed in warm water, mixed occasionally and equilibrated until they reached the desired temperature of 80° F. (26.7° C.). Sampling errors were minimized by taking a random and sufficiently large sample of peas of a narrow size range *(4, 11, 16)*.

Effect of evacuation. Complete evacuation of the entrapped air in submerged peas is difficult. With no evacuation, replicate measurements of the density of peas were highly variable. Removal of air in submerged peas by a mechanical vacuum pump for 4 or 5 minutes at 29 in. (30 mm. of Hg) gave satisfactory results. The evaporation of water from the sugar solution under vacuum causes an increase in its density and a drop of temperature, both of which may produce large errors, unless corrected. Evaporative cooling during evacuation was compensated by starting with a solution 2° F. above the desired temperature; density of the sugar solution was redetermined after each evacuation.

Selection of the liquid. With previously described methods for measuring density of frozen peas by liquid displacement in salt and other liquids *(4, 6, 7, 12)* the desired precision was not obtained. For example, it was found that a brief immersion (during the measurement) of peas in 0.5% sodium chloride solution appreciably increased through leaching the density of the solution, i.e. from 1.0008 to 1.0022. Various concentrations of sodium chloride solution up to 15% were tried and found unsatisfactory.

The above observations led to a search for a more suitable liquid. Serious problems were encountered in the use of several organic liquids and mixtures. Best results were obtained with salts of heavy metals (such as copper sulfate and lead acetate) and with sucrose solutions. A sugar solution was selected because it is non-toxic and is thus more suitable for use in a food laboratory.

Ideally, different concentrations of sucrose should be used for different maturity levels of peas; the concentration of immersion liquid should decrease as the maturity of peas increases to match the decrease in soluble matter in more mature peas. An 8% sucrose solution was used for all density determinations because its density changed least when it was infiltrated into peas of different maturities. Its density changes, for example, only by 0.0004 units in 70 minutes with peas of intermediate maturity (tenderometer 108). Furthermore, any density changes that did occur in the liquid were included in the calculations; the density of infiltrating sugar solution was determined at the end of each density measurement. At the same time, the observed density of the peas also changed relatively slowly, i.e. about 0.0004 units after 5 minutes of immersion in 8% sucrose solution (Figure 1).

[a] The subtraction of 0.0013 from the hydrometer reading at 80° F. (26.7° C.) converts this reading to density at 80° F. or 26.7° C./4° C. This hydrometer was made to read sp. gr. at 60° F. (15.6° C.). The hydrometer was carefully cleaned and dried after each determination.

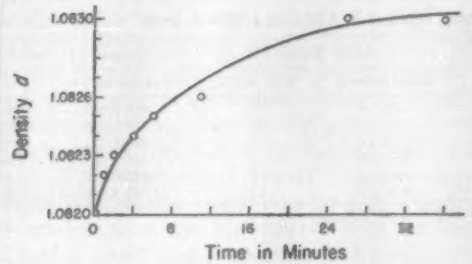

Figure 1. Change in density, d, of peas with time of submergence in an 8% sucrose solution at 26.7° C. (80° F.). Thomas Laxton peas, sizes 5 + 6, tenderometer value of raw peas 108.

The satisfactory behavior of the sucrose solution is probably due to the fact that the relatively large sugar molecules diffuse in or out of the peas rather slowly as compared, for example, to sodium chloride. The latter is known to be absorbed appreciably in commercial processing of peas *(1, 10)*.

This difference in behavior is striking even at very high concentrations of sucrose as shown in the results of a flotation experiment (Figure 2), where the behavior of peas in a 22.4% sucrose solution is compared with that in a 13% sodium chloride. The number of sinkers in 10 seconds was the same for both solutions; but in the salt solution the number of sinkers increased rapidly with time, whereas in the sugar solution the number remained practically constant for at least 390 seconds.

RESULTS AND DISCUSSION

The density method as described above was applied to 68 samples of peas representing a wide range of maturities, 4 varieties, and 3 seasons. The unevaluated results of these measurements were presented previously along with a number of other studies of the same peas *(8)*. Data on density have now been grouped and analyzed statistically (Tables 1, 2, and 3); the density results have been compared with organoleptic evaluation of maturity (cotyledon texture scores) and with percent alcohol-insoluble solids (percent A.I.S.).

Range of density values. The range of densities in peas of all grades, grouped by varieties and by harvest seasons is shown in Table 1. Although densities varied from 1.037 to 1.096, the corresponding tenderometer values of the raw peas ranged from 69 to 148, and the range of alcohol-insoluble solids (percent A.I.S.) of the same peas varied from 7.2 to 22.1. Percentagewise, a difference in density of 5.7% coresponds to an A.I.S. difference of 207% and a tenderometer reading change of 114%. These figures illustrate the narrowness of the range in terms of density values of pea samples which varied from immature grade A peas to grade D.

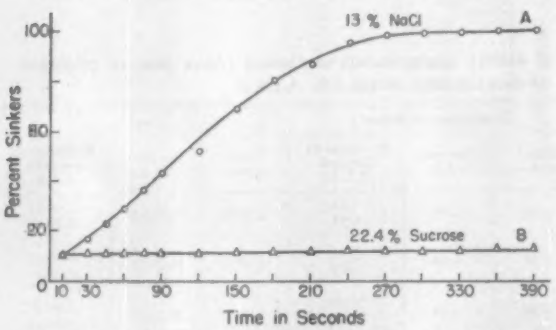

Figure 2. Change in percent sinkers with time (peas with skins removed).

A. 13% sodium chloride solution (NaCl).

B. 22.4% sucrose solution which gave the same number of sinkers at 10 seconds as 13% NaCl.

TABLE 1

Density (d) of peas by variety and season. Range of maturities from tenderer than commercial grade A to grade D [1]

Year	Variety	No. samples	Range of densities (d)		Average standard deviation Sw [2]
			Measuremets	Δd	
1949	Thomas Laxton	12	1.0373-1.0724	0.0351	0.0016
1949	Rondo	12	1.0385-1.0672	0.0287	0.0008
1950	Thomas Laxton	13	1.0481-1.0962	0.0481	0.0005
1950	Oracle	11	1.0487-1.0851	0.0368	0.0005
1951	Thomas Laxton	9	1.0506-1.0926	0.0420	0.0010
1951	Dark Seeded Perfection	9	1.0420-1.0931	0.0511	0.0008
				Av. 0.0403	Av. 0.00087
		Average Standard Deviation		0.0009	

[1] Corresponding range of tenderometer readings of raw peas was: 69-148.

[2] Sw = standard deviation estimated by method of Dean and Dixon *(2)*. Detailed data on individual samples are given in an earlier paper *(8)*.

In order to show the existing small differences in density between pea samples with certainty, high precision is required, as was indicated earlier. That such precision is not easily obtained is shown by the appreciable standard deviation values listed in Table 1. Accurate determinations of density are time consuming; although suitable for research purposes, other methods, not based on density, may be preferred for routine estimates of maturity *(8, 9)*.

Comparison of density with other measures of maturity. Table 3 shows that density measures the quality known as maturity in peas as the average density measurements correlate fairly well with average organoleptic cotyledon-texture scores and with the percent alcohol-insoluble solids. *Sxy* (standard error of estimate) values give the extent of predictability of average density values from the average cotyledon texture scores or from average percent A.I.S. *Sxy* serves better to compare methods than does *r* (coefficient of correlation) which is influenced by range.

Both the correlation coefficient *(r)* and standard error of estimate, *Sxy*, in density units, give a statistical comparison of density with cotyledon texture and with

TABLE 2

Distribution of densities (d) and percent alcohol-insoluble solids (% AIS) in grade A peas[1]

Year	Variety	No. of samples of diff. maturities	A.I.S. Range		Density *(d)* Range	
			Measurements	Δ % A.I.S.	Measurements	Δd[2]
1949	Thomas Laxton	8	7.2-11.8	4.6	1.037-1.060	0.023
1949	Rondo	9	7.2-12.5	5.3	1.038-1.057	0.019
1950	Thomas Laxton	6	7.6-13.2	5.6	1.048-1.071	0.023
1950	Oracle	6	8.1-12.5	4.4	1.048-1.069	0.021
1951	Thomas Laxton	6	8.0-12.3	4.3	1.051-1.067	0.016
1951	Dark Seeded Perfection	3	8.5-13.9	5.4	1.042-1.068	0.026

[1] The corresponding range of tenderometer readings of raw peas was 69-101; peas of size 2 were included.
[2] Av. range $\Delta d = 0.021$.

percent A.I.S. Here the average expected error of estimate is 0.0028 density units or about *7%* of the average range of densities (by varieties and seasons $\Delta d = 0.0403$ in Table 1), or about 13% of the average density range of grade A peas (Table 2).

Variation in density of peas with variety and seasons. Figure 3 shows that increases in density with increased maturity as measured by percent A.I.S. are of the same order of magnitude for all varieties and seasons. The steepest lines in Figure 3 are (1) Thomas Laxton (T.L.) peas for 1949 (slope = 0.0040 density units/percent A.I.S.) and (2) Dark Seeded Perfection (D.S.P.) for 1951 (slope = 0.0038 density units/percent A.I.S.), while all the others have nearly the same slope, i.e., 0.0030-0.0032 density units/percent A.I.S. The regression line for Rondo peas, though parallel to 4 others (same slope), was the lowest on the graphs; i.e., Rondo peas had the lowest density values for a given percent A.I.S.

The fact that differences in the slopes of the fitted lines in Figure 3 were relatively small shows that the density of peas of different varieties and seasons changed in much the same way as did their percent A.I.S. [an established measure of maturity *(5, 12)*]. In other words, Figure 3 supports the assumption that density in all varieties studied increases with maturity regularly and in nearly the same fashion. The absolute values of density, however, may differ appreciably for different varieties (or seasons) at any given percent A.I.S. or maturity level (Figure 3).

Relation of density to brine separation. Until now, the major causes of poor brine separation of peas in packing plant quality graders have been obscure *(1, 3, 13, 15)*. It is apparent from the above studies that the

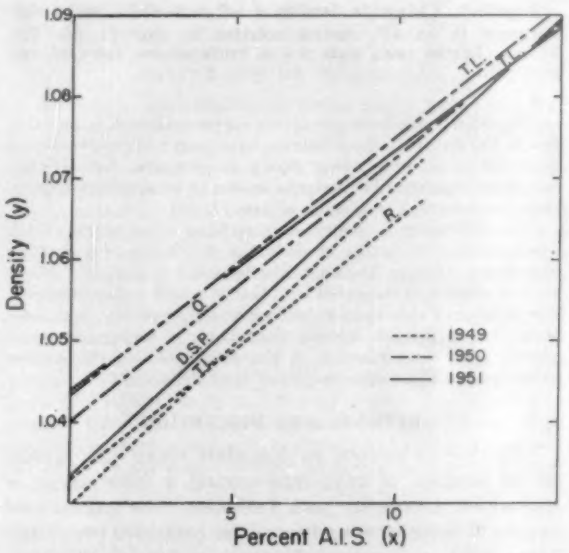

Figure 3. Variation in density (y) vs. % A.I.S. (alcohol-insoluble solids) (x), with varieties and seasons. The regression lines were fitted by the method of least squares as follows:

1949. T. L. (Thomas Laxton) — a = 1.00945; b = 0.00399; x = 7.2, y = 1.0382; x = 16.1, y = 1.0737.
1949. R. (Rondo) — a = 1.0163; b = 0.00324; x = 9.0, y = 1.0455; x = 15.0, y = 1.0649.
1950. T. L. (Thomas Laxton) — a = 1.0271; b = 0.00320; x = 10.0, y = 1.0591; x = 20.4, y = 1.0924.
1950. O. (Oracle)—a = 1.0240; b = 0.00325; x = 8.1, y = 1.0503; x = 19.2, y = 1.0863.
1951. T. L. (Thomas Laxton) — a = 1.0287; b = 0.00298; x = 8.0, y = 1.0526; x = 18.0, y = 1.0824.
1951. D. S. P. (Dark Seeded Perfection) — a = 1.0143; b = 0.00376; x = 8.5, y = 1.0463; x = 22.1, y = 1.0974.

TABLE 3

Correlation coefficient (r) and standard error of estimate (Sxy) of density measurements on thawed frozen peas vs. cotyledon texture scores (panel scores) and percent alcohol-insoluble solids (% A.I.S.)[1]

Year	Varieties	No. of samples	Correlation coefficient *r*			*Sxy*[2]		
			Panel scores	Percent A.I.S.	By varieties percent A.I.S.	Panel scores	Percent A.I.S.	By varieties percent A.I.S.
1949	Rondo	12	0.98	0.97	0.98	0.0025	0.0027	0.0022
1949	Thomas Laxton	12			0.97			0.0031
1950	Oracle	11	0.97	0.98	0.98	0.0034	0.0027	0.0024
1950	Thomas Laxton	13			0.98			0.0024
1951	Dark Seeded Perfection	9	0.98	0.98	0.99	0.0032	0.0029	0.0028
1951	Thomas Laxton	11			0.99			0.0014
	Average		0.98	0.98	0.98	0.0030	0.0028	0.0024

[1] Thomas Laxton (T.L.) peas were appraised organoleptically each year together with one of the other 3 varieties in a balanced incomplete block design *(8)*. All statistical values involving panel scores are based on both varieties for each year.
[2] *Sxy* = standard error of estimate in density units from cotyledon texture scores and from percent A.I.S.

narrowness of the density range in processed peas is an important factor in the difficulty of obtaining adequate separation or unequivocal grading by brine flotation methods. Another factor that contributes to this difficulty is the change in the density of the peas and of the brine solution during the flotation process. Temperature fluctuations may also contribute to these difficulties *(1)*. Yet, the process of flotation is correct in principle and is the only one available so far for separating the more mature from the less mature peas; it must be used until a better procedure can be substituted.

One improvement might be to use sugar solutions (sirups) instead of brine *(16)*; the practical details, including costs and sanitation problems, remain to be worked out. The facts presented in this paper may serve as a basis for future research leading to the improvement in processing equipment and method for the separation of commercial grades of peas. The use of sugar-sirup separation of peas and other frozen or canned foods would place such products in the category of salt-free-foods for the growing number of consumers requiring diets low in sodium.

SUMMARY

A reliable procedure has been developed for measuring the density of frozen peas by displacement in sucrose solution. Precision required for the characterization of peas of different maturities depends on proper control of conditions and adequate sampling of relatively homogeneous material.

The results show that the density of peas increases with advance in maturity. The density of peas of several varieties, seasons, and harvests correlated fairly well with panel appraisal of texture and with alcohol-insoluble solids of the same peas.

The range of densities was narrow for a wide range of quality grades used (A to D).

The small differences in density between grades and other sources of error explain many problems in the separation of peas by brine. The presented information may be helpful in further studies to improve industrial separation of peas of different maturities.

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An Apparatus Designed for the Rapid Electrochemical Estimation of Flavors In Vegetables[a, b]

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ULTIMATE PURPOSE OF THE RESEARCH here reported is the development of an apparatus which, at a series of differential electrodes, will characterize the flavors of vegetables quantitatively and qualitatively and do it very rapidly. This is, of course, a major problem and one which might seem to be essentially insolvable. Actual progress along such lines was made long ago, however, by the measurement of pH and total titrable acidity, which can be correlated with the subjective phenomenon of sourness. Sweetness, too, in special cases, can be estimated by objective means.

The present investigations have confined themselves strictly to the olfactory aspects of flavor, those due to the volatile flavoring constituents. In this field excellent work has been done by Ehrensvärd *(3)*, and at least two distinct approaches to the problem have been undertaken at Purdue University *(1, 2)*. For the most part, however, flavor quality, when it has been estimated objectively at all, has been determined indirectly, as by ascorbic acid content. Unfortunately, ascorbic acid level may at times be extremely poorly correlated with subjective reactions to flavor. This may be true even in vegetables where it is commonly used as an indication of edible quality.

It was not intended that the apparatus herein described should determine individual compounds nor that it should determine total oxidizable or total reducible volatiles at several oxidation-reduction potentials. Rather the general objective was that there should be developed a device analogous to such optical instruments as the Hunter Color and Color Difference Meter—that is, a device that can characterize a flavor, both for quality and quantity, by the pattern of reactions at a series of sensing elements. So much as is known of the anatomy, histology, and cytology of the olfactory mucosa seems to support the inference that the vertebrate animal may make its evaluations of flavors and odors with a series of different sensing elements electrically similar to those being worked on by the present investigators.

It is emphasized here that an objective test must ultimately be standardized against the subjective responses of consumers. Such standardization is obviously difficult in an area where subjective responses are often rather general and ill-defined. Nevertheless, the differential responses of different individuals should be amenable to statistical treatment and are therefore not so serious an obstacle as might be assumed. Some of the ultimate advantages of increased objectivity and quantitativeness in the estimation of flavor in vegetables have been pointed out by Hall, Marshall and Hartman *(4)*.

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[b] Presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 12, 1956.

CONSTRUCTION AND OPERATION

In a preceding paper *(5)* the senior author reported on qualitatively different responses at different microelectrodes. Subsequent work has been devoted largely (a) to the many problems of uniform presentation of the volatiles to the sensitive element and (b) to the greater refinement of the electronic system. The goal in each case was to get closely similar results from replicate tests.

Problems of contamination of the apparatus from water, from materials used in previous tests, or from air sources have been extremely troublesome. The difficulty is that, for many volatiles, the apparatus is apparently more sensitive than the human nose.

Construction. The sensitive element of the apparatus consists of a 20-gauge or smaller platinum wire dipping into an electrolytic solution. The other half-cell is a large platinum plate. The area of contact (with the solution) of this plate is at least a thousand times the area of the contact made by the sensitive element wire. Voltages, ranging from 3.0 to 0.0, either negative or positive, are impressed on the electrolytic cell and the resultant current measured. In the microelectrode or sensitive element most commonly used, the end of the platinum wire is insulated from the solution by a glass tip presumably thick enough (approx. 0.5 mm.) to prevent the ready flow of electrons or the migration of hydrogen ions through the tip. The dipping of the electrode is regulated by a micrometer in such manner that its depth is closely controlled. Figure 2 is a photomicrograph of the physical relations between the solution and the glass-tipped microelectrode.

Procedure. Contamination from preceding samples was fairly well eliminated by washing and baking the sample-containing tubes, the diminutive beakers inside the syringes and the delivery tubes. Baking consisted of heating the glassware for approximately 12 hours at 110° C. or a higher temperature.

In running a test, the odorant material was placed in a shallow glass beaker (a 30-ml. beaker cut down to a total height of 1.0 cm.) on the top of the plunger within the glass syringe (B, Figure 1). As much air as possible was expelled from the syringe before it was fastened in its cradle. The beaker itself prevented the plunger from moving to the top of the cylinder. Then, outside air, fairly free of odors, was drawn into the syringe by attaching the syringe tip to the intake tube (K, Figure 1) and drawing down the plunger either by hand or with the motor-driven screw.

After a two-minute volatilization period, the plunger was pushed steadily upwards by the motor and the air and volatile chemical mixture expelled fairly slowly against the sensitive microelectrode; the microelectrode had previously been chemically polarized. In the tests here reported the microelectrode and the large platinum reference electrode were electrically connected through a single solution. The large electrode was immersed; the very small one was barely in contact. The polarization mentioned above was accomplished by impressing a D.C. potential on the electrolytic cell until the current in the circuit decreased to a constant low level. The potential for polarization was the one also used in the particular test.

In most cases, the effect of the vapor on the microelectrode was to increase the current; in some cases, however, a momentary or persistent decrease was obtained. In general, the re-

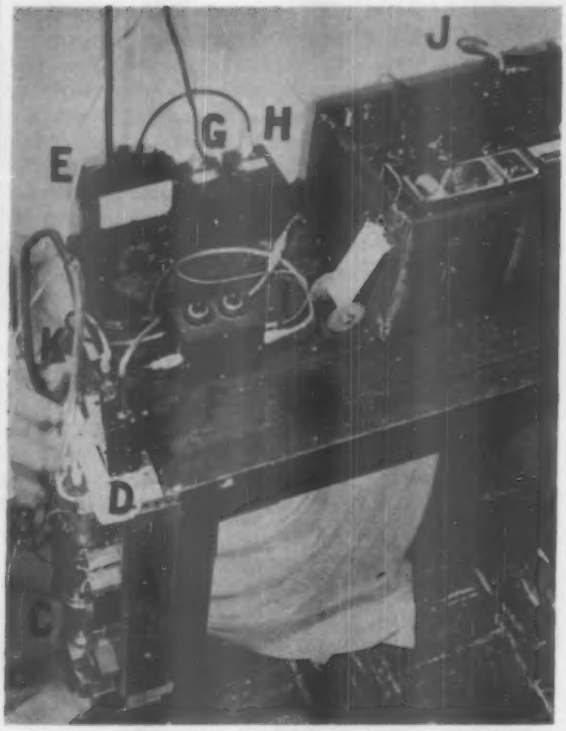

Figure 1. Apparatus as a Whole. A—Section designed to present vaporized odorant to electrodes. B—One of many interchangeable glass syringes heated by thermostatically controlled jacket adjusted to give volatilized odorants in air the temperature of the human body. C—Motor-driven screw made to expel odorants in air from the syringe at about 1.23 ml. per second. D—Electrically shielded box containing sensitive microelectrode and large platinum plate dipping in the same electrolyte as the microelectrode. Temperature of this box is also ordinarily held at 37° C. E—Weston voltmeter recording constant potential impressed on the electrolytic system. F—Helipot potentiometers for regulating voltage available from dry cells G or H. I — Shielded leads. J — Millivac Micro-Micro-Ammeter Recorder, modified for convenience in operation and for minimizing the effects of extraneous static electricity and changes in power supply. This recorder is sometimes replaced by other electrical measuring devices. K—Piped-in source of outside air.

sponse was characteristic of the chemical. All these changes were recorded on tape by the Millivac Micro-micro-ammeter.

RESULTS

Objective data have been obtained on only a small number of possible combinations of variables which the instrument easily permits to be varied. Much time has been spent in standardizing the equipment, devising means of avoiding contaminations, and, in general, developing the instrument so that it will give reproducible results.

An example illusrating typical reproducibility of results is shown in Figure 3. Sometimes replicates were much poorer than these, but many were so nearly the same that they might almost have been photographically reproduced one from the other.

The shape of the amperage-time curve obtained on the apparatus was often typical of the odorant. Originally it was thought that no attention need be given to these forms and that the chief numeral of interest would be the maximum sustained response to the odorant. This viewpoint may have to be modified.

On the basis of maximum sustained response, data such as those included in Table 1 were made up for a number of odorants. Most of these chemicals, perhaps all of them, contribute characteristic flavors to vegetables or fruits. Therefore, they should serve well as standards in exploring the areas of response. The data in Table 1 represent results from only two of the almost innumerable sensitive element combinations that are possible. They are the two with which the most accurate work has been done by the present investigators. A study of Table 1 illustrates the very different reactivities of different volatilized substances at the same electrode and of the same substances at different electrodes.

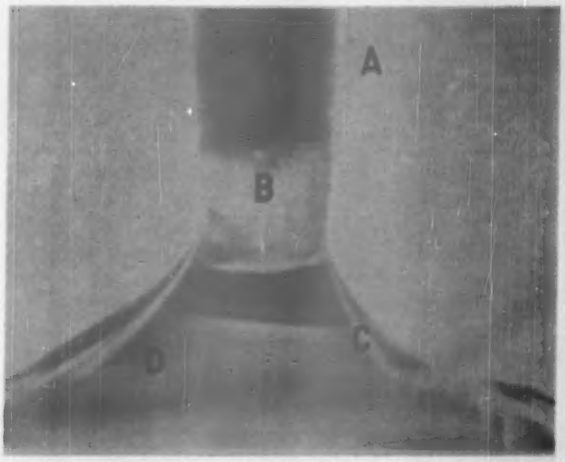

Figure 2. Polaroid Camera Photomicrograph of Microelectrode Dipping in Buffer Solution. A—Platinum wire. B—Glass tip of microelectrode. C—Buffer solution. D—Vague outline of end of glass tube containing solution. The microelectrode in this photograph was immersed less deeply than was usual in order to show glass tip. Shape of film and other features remain about as shown, regardless of immersion depth. Magnification is approximately 26 ×.

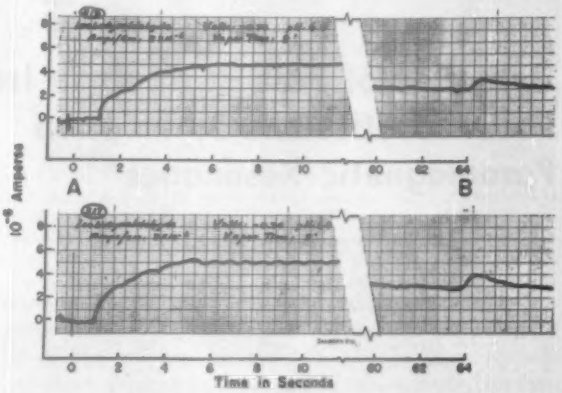

Figure 3. Typical duplicate amperage-time curves produced on micro-micro-ammeter recorder when air presumably saturated with isobutyraldehyde was passed over the sensitive element. The middle parts of the graphic galvanometer tapes have been cut in order that the more important features might be shown on a larger scale. A marks the time when the current of air and odorant first struck the sensitive element; B, the time when the current of air and odorant was shut off.

DISCUSSION

Although the results on which this paper is based were obtained with a bare, glass-tipped microelectrode and on sodium acid phosphate solutions at pH values from 5 to 8, these conditions are only a few of the many which might have been used. Electrodes of various

TABLE 1

Reactions of several odorants (volatile flavoring constituents) at two microelectrode conditions[1]

Volatile flavoring constituent (allowed a 2-minute volatilization period)	Responses to odorants	
	For + .70 Volt D.C.	For − .70 Volt D.C.
Allyl sulfide	+ 59.5 ± 5.5	− 120.0 ± 0.0
Ethyl-n-butyrate	+ 2.1 ± 0.3	0.0
Acetic acid	+ 149.0 ± 13.6	+1275.0 ± 175.0
Pyridine	+ 30.0 ± 0.0	Doubtful
Di-n-propylamine	+1425.0 ± 75.0	− 13.3 ± 6.7
Ethyl alcohol	+ 95.0 ± 5.0	Doubtful
Ethyl acetate	Doubtful	0.0
d-Limonene	+ 463.3 ± 18.5	+ 310.0 ± 30.0
Isobutyraldehyde	Erratic	+4933.3 ± 176.4

[1] Sensitive electrode was 20-gauge platinum wire with a glass tip. Buffer: Na_2HPO_4 — NaH_2PO_4 mixture at pH 6.0. Potential impressed upon test cell system is indicated. Readings and standard errors are all in millimicroamperes; positive figures indicate an increase in current; negative ones, a decrease.

shapes and sizes, coated with many substances but especially with water-insoluble ones, have been tried to a limited extent. They will be tested much more extensively in future experiments. Solvents and solutes, especially organic ones, may be widely varied. In the almost unlimited possibilities of substitution of essential parts seems to lie the promise of the instrument.

At present, there is no assurance that the apparatus will be able to discriminate among all vegetable flavors. But the data indicate that where the number of distinctive or characteristic odorants may be very limited, the method can be used to determine very quickly the extent to which a given vegetable sample departs from an objective standard established by this apparatus.

At the present time the investigators tentatively conclude that the principal effects being recorded are due to oxidations and reductions of the volatile compounds or their products at the microelectrode. Experiments have seemed strongly to indicate that changes in surface tension and those due to the mechanical disturbance of the surface of the liquid around the microelectrode are of only secondary importance. They are of the opinion that the microelectrode dipping in an electrolyte is closely analogous in structure and function to olfactory hairs protruding from the olfactory mucosa in the nose.

SUMMARY

Rapid and reproducible responses to many volatile flavoring constituents of vegetables were obtained with the apparatus used. Different responses were obtained with different odorants at a single type of sensitive element polarized at a specific voltage. The pattern of response to the series of chemicals changed greatly when the conditions at the sensitive element were changed.

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Detection of Free Radicals in Irradiated Food Constituents by Electron Paramagnetic Resonance[a, b]

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(Manuscript received August 16, 1956)

Free radicals are thought to be responsible for many of the undesirable side effects (off-flavors, color changes, and vitamin destruction) encountered in irradiated foods at sterilizing doses of ionizing radiation. Until recently, methods for the direct detection and identification of free radicals in irradiated foods and food constituents have not been available, and the existence of radiation-induced free radicals has been inferred from the stable reaction products which they produce in certain chemical systems. Although much has been learned about the nature of these free radicals by such indirect methods, a means for the direct detection of the free radicals themselves in complex systems has long been needed.

The recently developed technique of electron paramagnetic resonance (EPR) provides a powerful method for detecting free radicals *in situ;* and it promises to be useful for determining the nature of the free radicals produced in irradiated foodstuffs, and for studying the environmental factors which affect their yield and stability. This paper presents some preliminary results of electron paramagnetic resonance studies on irradiated foodstuffs and food constituents.

[a] Based on material presented at the Sixteenth Annual Meeting of the Institute of Food Technologists, St. Louis, Missouri, June 13, 1956.

[b] This work was performed under Contract No. DA19-129-QM-378, Quartermaster Corps, Department of the Army, and has been approved for publication.

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THEORY OF THE ELECTRON PARAMAGNETIC RESONANCE METHOD

For a detailed presentation of the theory of electron paramagnetic resonance, the reader is referred to the excellent review by Shoolery and Weaver *(6)*. The papers of the recent Faraday Society symposium on microwave and radiofrequency spectroscopy *(5)* provide an introduction to current experimental work in this field.

Briefly, the method to be described takes advantage of the fact that free radicals contain at least one unpaired electron per radical, and that unpaired electrons have unique magnetic properties which permit their detection by magnetic resonance techniques. The EPR method is based on the resonance absorption of electromagnetic energy when a substance containing unpaired electrons is simultaneously exposed to a radiofrequency field and a strong polarizing magnetic field, the polarizing field being oriented perpendicularly to the radiofrequency field. The condition for resonance absorption is given by

$$f = \frac{g\mu_B H_o}{h} \qquad (1)$$

where f is the frequency of the radiofrequency field; g is the spectroscopic splitting factor of the unpaired electron; μ_B is the Bohr magneton; H_o is the strength of the polarizing magnetic field; and h is Planck's constant.

Since the unpaired electrons in most free radicals have "g" values very nearly the same as that of the free electron, this expression can be reduced to the approximate equation

$$f = 2.8\, H_o \qquad (2)$$

where f is now the frequency of the radiofrequency field in megacycles per second and H_o is the strength of the polarizing field in gauss.

The majority of the work to date on the detection of free radicals by EPR has been carried out at microwave frequencies, usually at about 9,000 mc., which is in the familiar 3-cm. band of World War II radar. The corresponding polarizing magnetic field of about 3,000 gauss is easily obtained with a good laboratory electromagnet. In Figure 1 is shown a block diagram of a typical microwave paramagnetic resonance spectrometer. The radiofrequency field is provided by the microwave Klystron oscillator. The sample is placed in a microwave cavity resonator which is located between the poles of an electromagnet. A crystal detector is used to measure the amount of microwave power absorbed by the sample. Various additional electronic devices provide a means for graphically recording the rate of change of the microwave absorption of the sample as a function of varying magnetic field, the frequency being held constant. The principal advantages of the microwave electron paramagnetic resonance technique are that it is very rapid, it is entirely nondestructive, it can be applied to opaque liquids, solids, and gases, and it is essentially free from interference from substances other than free radicals.

Previous work. The present study was undertaken as a result of the successful detection of the free radical, atomic hydrogen, in irradiated ice by Smaller of the Argonne National Laboratory *(7)*, using the electron paramagnetic resonance technique. Smaller also found some evidence of the hydroxyl radical, OH. Almost simultaneously, Livingston *(4)* reported the detection

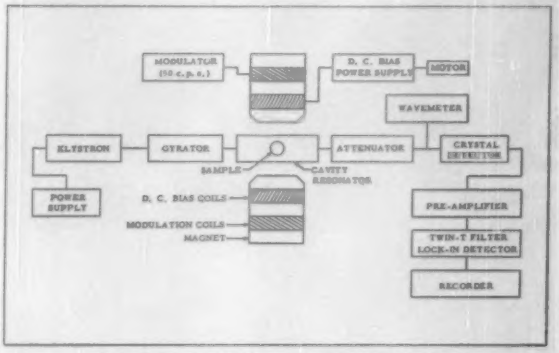

FIGURE 1. MICROWAVE PARAMAGNETIC RESONANCE SPECTROMETER

Figure 1. Block diagram of microwave electron paramagnetic resonance spectrometer.

of atomic hydrogen in various acids irradiated at liquid nitrogen temperatures. Although the lifetimes of the primary radiation induced free radicals are much too short in aqueous solutions for detection by this technique, Smaller and Livingston were able to build up a sufficiently high steady state concentration by irradiating the samples at liquid nitrogen temperatures, thus preventing recombination of the free radicals.

Free radicals have since been observed in irradiated carbohydrates by Combrisson and Uebersfeld *(1)*, and in irradiated proteins and amino acids *(2)* and carboxylic and hydroxy acids *(3)* by Gordy *et al.*

EXPERIMENTAL

Equipment. In Figure 2 is shown the microwave electron paramagnetic resonance spectrometer used in this investigation. The apparatus is of conventional design *(8)*. It employs a 2K25 Klystron operated at a nominal frequency of 8800 megacycles per second, a transmission type microwave cavity resonator, a crystal detector, a "lock-in" amplifier for demodulation of the 50 cycle absorption signal, and an Esterline-Angus recorder. For most of the measurements, the 2K25 Klystron was operated at maximum microwave power output. The polarizing magnetic field of about 3140 gauss was provided by a Varian Model 4004 electromagnet. The sensitivity of the EPR spectrometer was estimated to be about 2×10^{14} unpaired electrons in a 200 mg. sample.

The irradiations were carried out in a cylindrical 160-curie Cobalt-60 irradiation source which provides a uniform radiation field of approximately 340,000 rep/hr. over a source volume ⅝ inch diameter by 4 inches long.

Samples. Most of the samples were irradiated as powders on an "as is" basis, i.e., without further purification or drying. The pure carbohydrates and amino acids were of reagent grade, purchased from commercial sources. Certain of the starches and proteins, and some electron-irradiated carbohydrates,* were obtained from private sources. Some experiments were carried

* Kindly supplied by Professor M. L. Wolfrom of The Ohio State University, Columbus.

Figure 2. Microwave electron paramagnetic resonance spectrometer.

out on materials in which the moisture content had been adjusted, either before or after irradiation.

Samples were irradiated in sealed 9 mm. test tubes and in sealed 3 mm. capillary tubes. Usually, no attempt was made to remove oxygen from the samples prior to irradiation. After irradiation, the samples were transferred to unirradiated capillary tubes, sealed, and stored at room temperature. The post-irradiation transfer was necessary because irradiated Pyrex shows a moderate EPR absorption. Later it was found possible to seal small samples in the bottom of 9 inch capillary tubes, irradiate them, and then invert the tubes for the EPR measurements. No EPR absorption was obtained from the portion of the tube that extended outside the source. In most cases, the EPR measurements were made within a few hours after removal of the samples from the source. About 20 mg. of material was usually sufficient for the measurement. EPR measurements have been repeated on some of these same sealed samples at intervals for periods of up to 8 months' storage at room temperature.

RESULTS AND DISCUSSION

A large number of carbohydrates, amino acids, proteins, and miscellaneous food items have been found to exhibit electron paramagnetic resonance absorption after irradiation as dry solids with varying doses of Cobalt-60 gamma radiation. At doses of 1 to 25 megarep, strong EPR spectra have been obtained from most of the substances examined. EPR spectra have been found in some substances at radiation doses as low as 125,000 rep, and it is probable that with improved instrumentation, the limit of detectability can be extended well below this dose level. The free radicals in many substances are surprisingly stable, and EPR spectra can still be observed after several months' storage at room temperature.

The free radicals examined by us so far appear to have "g" values very close to that of the free electron. The "g" value of irradiated mannitol, for which precise measurements have been made, is within 0.1% of that of the organic free radical, diphenylpicrylhydrazyl, which was used as a reference standard.

Since the yield and stability of radiation induced free radicals have been found to depend on environmental factors during and after irradiation, absolute free radical yields were not measured precisely. An approximate value was obtained for anhydrous dextrose, a compound which appeared to have the highest free radical yield of the substances examined. When examined 8 days after irradiation with a dose of 1 megarep of gamma radiation, it was found to contain approximately 4×10^{17} free radicals per gram, based on its EPR absorption intensity compared with that of a weighed amount of diphenylpicrylhydrazyl.

EPR spectra may have several components because of the influence of internal magnetic and electric fields within the molecules on the unpaired electron. The recorded EPR spectrum of irradiated histidine dihydrochloride, an amino acid which has a particularly complex EPR spectrum, is shown in Figure 3.

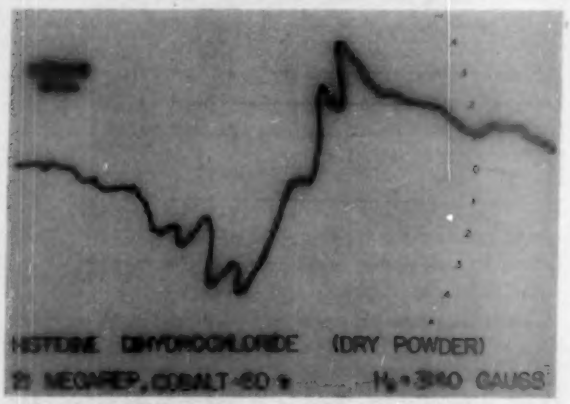

Figure 3. EPR spectrum of irradiated histidine dihydrochloride.

The fact that the EPR spectra from different irradiated substances are unique indicates that effects due to different chemical environments are being observed. It is of interest that free radicals produced by quite a different means as, for example, by the pyrolysis of carbohydrates, are not at all like those produced by ionizing radiations. In Figure 4 are shown the recorded EPR spectra of dextrose charred at about 300° C. for 30 minutes and of irradiated dextrose. The charred dextrose gives a simple, relatively narrow EPR spectrum, without any fine structure, while the irradiated dextrose gives a much broader spectrum with several components. A single line is to be expected for the free radicals resulting from broken carbon bonds in carbonized dextrose, since the environment of the unpaired electron probably consists almost entirely of carbon atoms. The unpaired electron in irradiated dex-

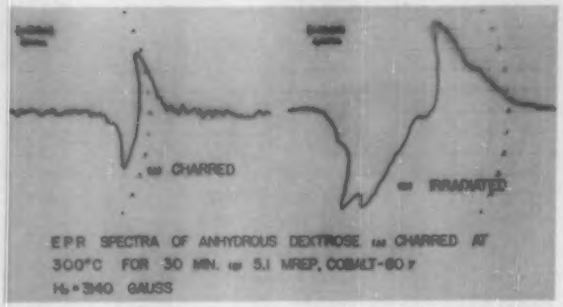

Figure 4. EPR spectra of charred (a) and irradiated (b) anhydrous dextrose.

trose is surrounded by various combinations of carbon, hydrogen, and oxygen, and its EPR spectrum reflects this environmental heterogeneity. It is also possible that in some cases the observed EPR spectrum arises from unpaired electrons in more than one position in the molecule.

The richness of spectral detail obtained in many irradiated substances is most striking, and it appears that there are possibilities for using the EPR technique along with irradiation, as an analytical tool.

In Figure 5 are shown EPR spectra from "Pream," a dried milk product, casein, and cornstarch. These are representative of the types of spectra obtained from over 40 irradiated substances examined thus far.

Effect of radiation dosage: The intensities of the EPR spectra appear to be proportional to total radiation dose over a fairly wide range. A study of the dose response is often complicated by the fact that free radicals in irradiated substances may have varying degrees

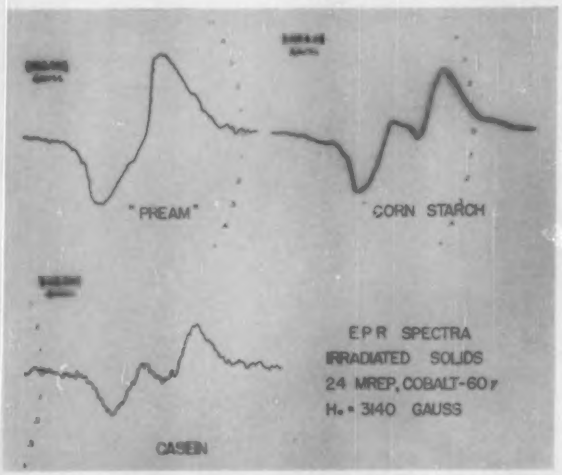

Figure 5. EPR spectra of irradiated "Pream" (a), casein (b), and cornstarch (c).

of stability, and when long irradiation periods are required to produce a large total dose, as is the case with the usual Cobalt-60 source, some loss of free radicals may occur before the final total dose is reached. Obviously, it would be desirable to conduct such a study with a high intensity radiation source such as a Van de Graaff generator, since the dose rate could be varied so that all doses could be delivered in the same short time period.

The apparent linearity of EPR intensity with doses over a range of 0.1 megarep to at least several megarep suggests the use of EPR for radiation dosimetry. Further work needs to be done to find a free radical with sufficiently long term stability, and EPR instrumentation needs to be improved so that intensity measurements can be made with greater accuracy than is now possible.

Factors affecting free radical stability: The relatively long time stability of radiation-induced free radicals in solids appears to be due, in large part, to their lack of mobility in the solid state. When irradiated sugars and amino acids are dissolved in distilled water and then redried over P_2O_5 at room temperature, no EPR spectra are found.

Heating an irradiated substance might also be expected to destroy the free radicals, and it was found that heating a sealed sample of irradiated glycine crystals for only a few minutes in a boiling water bath resulted in complete disappearance of the EPR spectrum.

As indicated earlier, the complexity of the EPR spectra from some irradiated substances may be due to the existence of unpaired electrons at several relatively stable sites in the molecule. Some evidence for this has been found by observing the gradual decay of the complex EPR spectrum of irradiated anhydrous dextrose. In Figure 6 are shown the EPR spectra from a single irradiated sample, measured over a period of several days' storage at room temperature. One of the satellite lines appears to fade more rapidly than the others, and the central portion of the spectrum becomes resolved, possibly indicating that one position of the unpaired electron is less stable than the others, or that a rearrangement to a more stable site is taking place.

Effect of moisture on free radical stability: Since radiation-induced free radicals disappear on dissolving an irradiated substance in water, it is of interest to determine free radical stability in moist solids over a wide moisture range. A series of unmodified cornstarches

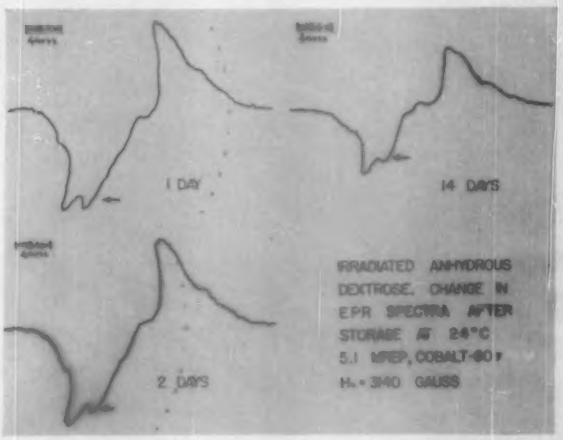
1 DAY
14 DAYS
2 DAYS
IRRADIATED ANHYDROUS DEXTROSE. CHANGE IN EPR SPECTRA AFTER STORAGE AT 24°C
5.1 MREP, COBALT-60 γ
H_0 = 3140 GAUSS

Figure 6. Change in EPR spectrum of irradiated anhydrous dextrose after storage at 24° C.

having moisture contents ranging from 3.2% to 14.5% was irradiated to a total dose of 24.5 megarep, and the EPR spectra were measured immediately after completion of the irradiation. The effect of moisture content was very striking, the spectrum from the 3.2% moisture content sample being very intense, while the EPR intensities from the higher moisture samples were progressively weaker. Moisture content also affected the rate of disappearance of the free radicals on storage at room temperature, the fading being most rapid at the higher moisture contents. Because of the pronounced decay of free radicals at high moisture contents, it would be desirable to irradiate the samples in a high intensity source in order to determine the "true" effect of moisture content before appreciable fading of the resonance occurs.

The marked effect of moisture content probably explains many of the differences in signal intensity observed in the large number of carbohydrates and amino acids irradiated in our first experiments, since no precautions were taken to adjust them all to a uniform moisture content. It probably also explains the absence of EPR signals in some irradiated substances investigated in other laboratories. From these results, it appears that it would be necessary to dry all samples thoroughly prior to irradiation, if comparable results are to be obtained.

The effect of absorption of additional moisture after irradiation was also determined. A batch of starch at 3.2% moisture was irradiated to a total dose of 24 megarep; then portions were equilibrated to various moisture contents. Again, the effect of moisture was striking. After about 4 days, during which time the samples equilibrated, the EPR signals remained very intense below 3% moisture, but faded to less than 10% of the initial intensity at 6% sorbed moisture, and disappeared entirely at 14.8% moisture.

CONCLUSIONS

Preliminary results have been presented to show how electron paramagnetic resonance techniques can be used for the *in situ* detection of free radicals in irradiated substances. The electron paramagnetic resonance arises from unpaired electrons in the free radicals, and the shape and width of the EPR signals reflect the chemical environment of the unpaired electron in the molecule. The observed free radicals appear to be produced by the direct action of the ionizing radiation on the dry constituents of the sample. It is doubtful that any of the EPR spectra observed in these experiments could have been due to atomic hydrogen or the hydroxyl radical. The stability of the free radicals is strikingly dependent on moisture content, and in very dry solids, the free radicals may be stable indefinitely at room temperature. Future experiments are planned to investigate more thoroughly other environmental factors which affect the yield and stability of free radicals during and after irradiation, and to extend the scope of this study to frozen systems in which less stable free radicals can be trapped for a sufficiently long time to permit their detection.

Acknowledgments

The authors are pleased to acknowledge the valuable assistance of Mr. George J. Slocum, electronics technician, Southwest Research Institute, in the maintenance and operation of the EPR equipment.

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SELECTED ABSTRACTS

(Continued from page 36 preceding technical papers)

The life span and the cause of death of inbred C_3H strain mice was modified markedly by the dietary regimen. Caloric restriction or an increase of vitamin E reduced the incidence of heart failure in old age from a semi-purified diet contg. a modified Jones-Foster salt mixt. Vitamin B_{12} postponed the age of appearance of this metabolic disorder. Caloric restriction and vitamin B_{12} seemed effective in preventing or delaying convulsive deaths, while a Crisco diet induced a high incidence of fatal convulsions.

Food processing and nutritive value for man.

BARNELL, H. R., AND HOLLINGSWORTH, D. F. (Great Westminster House, Horseferry Road, London). *Proc. Nutrition Soc., 15,* 142-7 (1956).

A review of the effects of processing on the nutrients in food must leave an impression of losses of nutrients whenever a food is given any form of treatment. On the whole this is true, but it must be emphasized that the processing of foods is to a large extent a necessity to make them readily available, edible and palatable.

PHYSIOLOGY AND MEDICINE

Cytochrome oxidase activity and body weight in rats and in three species of large animals.

KUNKEL, H. O., SPALDING, J. F., de FRANCIS, G., AND FUTRELL, M. F. (Texas Agr. and Mechanical Coll. System, Coll. Sta., Texas). *Am. J. Physiol., 186,* 203-6 (1956).

In the comparison of rats, sheep, swine and cattle, the cytochrome oxidase activity of skeletal muscle (principally of the gracilis) was found to be roughly in inverse function of body wt. The data support the concept that the summated total measurable cytochrome oxidase activity is very nearly proportional to body wt. to the 0.75 power and, hence, to basal metabolism.

Rate of elimination of labeled carbon dioxide from the body.

DRURY, D. R., WICK, A. N., AND ALMEN, M. C. (Univ. Southern California, Los Angeles). *Am. J. Physiol., 186,* 361-4 (1956).

When C^{14}-labeled bicarbonate is injected intravenously, about 10% of it is exhaled within 30 sec. by the lungs before it is mixed with body CO_2. Labeled CO_2 that has escaped elimination by the lungs is mixed with body CO_2 in a complex manner. The manner of elimination of labeled CO_2 produced in metabolism is also complex and does not simulate that following intravenous or intraperitoneal injection of labeled bicarbonate.

FOOD AND FOOD TECHNOLOGY

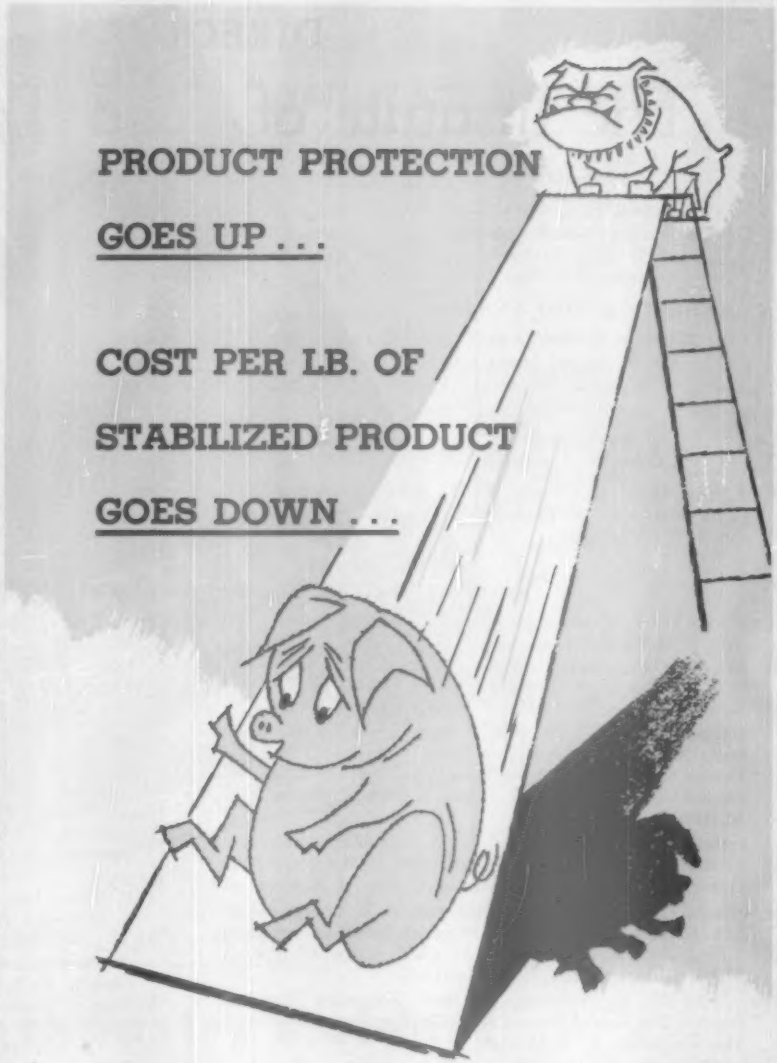
COFFEE AND TEA

The examination and tasting of tea.

Coffee and Tea Ind., 79, 47 (8)+ (1956).

Desirable liquor characteristics, such as

(Continued on page 42)



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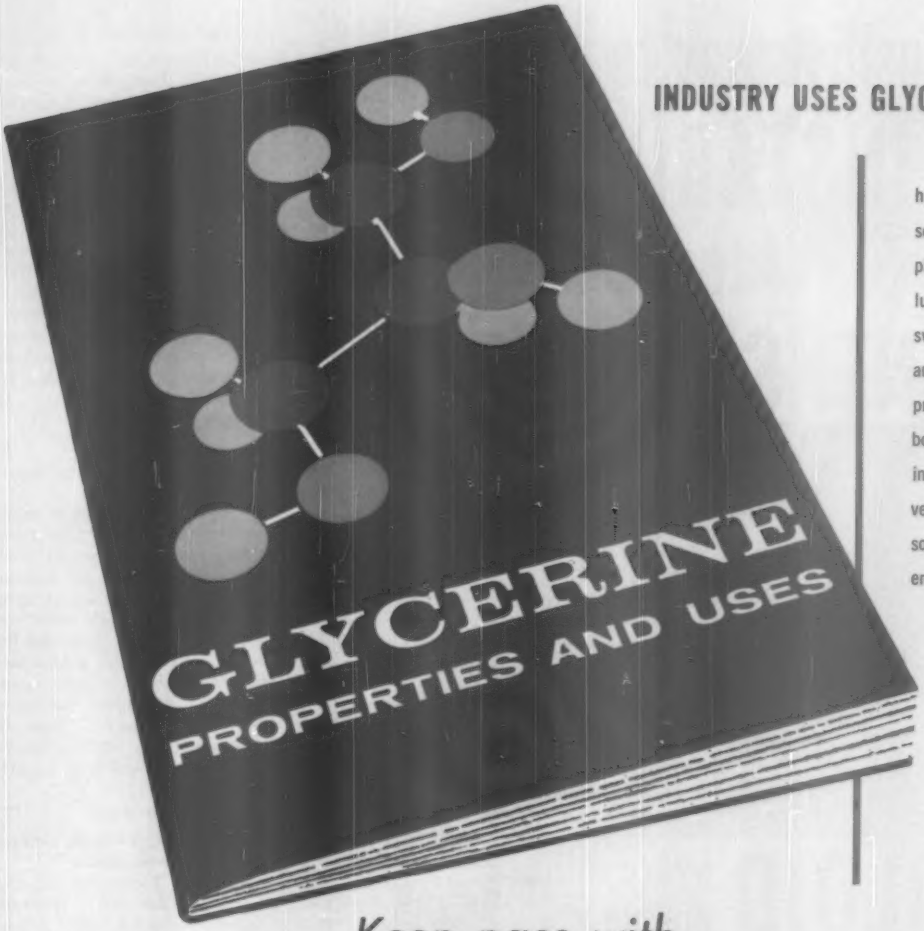
Write to nearest regional secretary, if within an area covered by a regional section, or to the Executive Secretary of the Institute for an application form and information regarding qualifications and classifications.



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(Continued from page 39)

quality, flavor, briskness, and strength are described. To retain these characteristics, tea should be packed with a moisture content between 3-4%.

FATS AND OILS

An improved system for the collection of essential oils.

HILDEBRAND, R. P., AND SUTHERLAND, M. D. (Univ. of Queensland, Brisbane, Australia). *Chem. & Ind.* (1956) 838-9.

The authors describe a simple modification by which the cohobation principle may be applied to direct steam-injection type leaf stills, resulting in improved yields of oil without addnl. steam consumption.

FEEDSTUFFS

Protein delinquency.

BIRD, H. R. (Univ. of Wisconsin). *Feed Age, 6* (11), 34-9 (1956).

A criticism of the formulation of poultry feeds stressing the need for increasing the level of effective protein in the feed. Methods of reaching amino acid balance are discussed. Methionine, a synthetic product, although being much more expensive than protein, is recommended for addn. to poultry diets because it improves the utilization of a relatively large quantity of protein. In the future the waste due to "protein delinquency" may be eliminated entirely when other synthetic amino acids become available at suitable prices.

MEAT AND FISH

Some chemical and organoleptic changes in gamma irradiated meats.

GRONINGER, H. S., TAPPEL, A. L., AND KNAPP, F. W. (Univ. of California, Davis). *Food Research, 21,* 555-64 (1956).

Irradiation produced significant organoleptic changes and destroyed thiamine and the carotenoid pigment of salmon. Formation of lipid peroxides and carbonyls was greatly reduced when radiation was carried out in an inert atm. or vacuum. At the sterilizing dose the succinoxidase system was still active in beef, port and tuna.

Chemical changes in fish actomyosin during freezing and storage.

SEAGRAN, H. L. (U. S. Fish and Wildlife Service, Ketchikan, Alaska). *Food Research, 21,* 505-9 (1956).

Freezing and thawing of actomyosin resulted in a very slight increase in reactive –SH groups in fraction sol. in dil. salt soln. The aggregated fraction showed a larger increase. Total –SH groups remained const. Intrinsic viscosity decreased immediately after freezing but did not change on storage. Actomyosin soly. increased to a max. after one month's storage and then decreased.

MILK AND MILK PRODUCTS

Methyl sulfide and the flavor of milk.

PATTON, S., FORSS, D. A., AND DAY, E. A. (Pennsylvania State Univ., Univ. Park). *J. Dairy Sci., 39,* 1469-70 (1956).

The paper presents preliminary evidence that milk contains the volatile product, Me sulfide, b.p. 38° C. The occurrence of abnormally high concns. of

(Continued on page 44)

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The old saying, "The proof of the pudding is in the eating thereof," certainly applies to our *complete* cake mix flavor. For as we continue our second year of advertising, this product is an established success.

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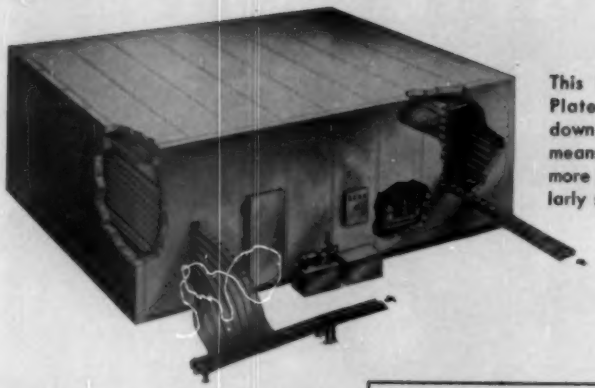

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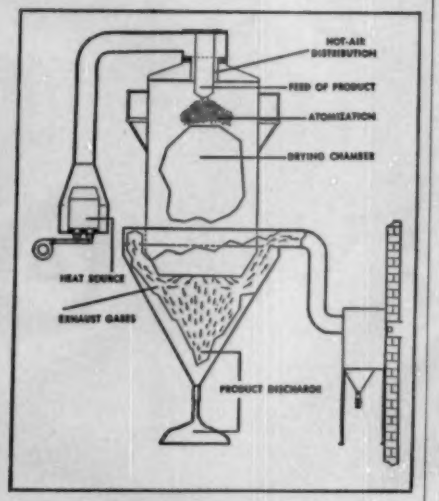

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(Continued from page 42)

Me sulfide could account for certain "cowy" and feed-type off-flavors, whereas abnormally low amts. of the compd. such as might be met in concd. or dried milk products may be responsible in part for the lack of so-called "fresh milk flavor."

The influence of environment and processing on spoilage organisms in cottage cheese.

HARMON, L. G., AND SMITH, C. K. (Michigan Agr. Expt. Sta., East Lansing). *J. Milk and Food Technol., 19,* 252-9 (1956).

Improperly pasteurized milk and cream, coagulator, starter, H_2O, air and contaminated equipment were found to be sources of spoilage organisms. Samples of several of the above caused spoilage when inoculated into sterile cheese and incubated at 21° C. for 4 days. Several H_2O supplies were found which contained high populations of lipolytic and proteolytic organisms, but were free of coliform bacteria which are commonly used as an index of acceptability for H_2O supplies. The varied sources of spoilage organisms indicated the need for observance of proper pasteurization procedures, equipment sanitation, precaution in handling raw materials and necessity for proper bactericidal treatment of wash H_2O.

Coconut-like flavor defect of milk fat. II. Demonstration of δ-decalactone in dried cream, dry whole milk, and evaporated milk.

KEENEY, P. G., AND PATTON, S. (Pennsylvania Agr. Expt. Sta., Univ. Park). *J. Dairy Sci., 39,* 1114-19 (1956).

Coconut-like flavor exts. were obtained from dried cream, dry whole milk, and evapd. milk. The presence of δ-decalactone in these flavor exts. was established by paper chromatography, and the characteristic coconut-like flavor defect of these products is attributed to this lactone.

POTATOES

Suppression of the sprouting of potatoes by the vapor of alcohols.

BURTON, W. G. (Ditton Lab., Larkfield, Maidstone, Engl.). *Nature, 178,* 218 (1956).

Alcohol vapors in general retard sprout growth in potatoes.

SUGAR, SIRUP AND STARCHES

Drying of starch by high frequency.

MAES, E., AND PIETERMAAT, F. P. *Rev. fermentations et inds. aliment., 11,* 106-10 (1956); *Chem. Abstr.,* 14251f (1956).

The detn. of moisture by the application of high frequency is described. The method has many advantages for the starch industry. The detn. requires only 15 min. for completion, and it can be applied to various substances including gluten feed, dextrin, and dextrose as well as to starch. The precision is equiv. to that of the drying oven. High sugar concns. cannot be successfully dried by this method.

Time of loading and strain retardation in starch jelly candy.

(Continued on page 46)

How to protect your foods against down-grading by using pure Diamond Crystal Flake Salt

Diamond Crystal *Flake* Salt has the highest uniform purity of any salt made. Ends danger of costly spoilage and "off" flavor from an impure granular salt or rock salt brine. Improves taste and keeping qualities of foods.

DO YOU KNOW what the calcium, magnesium, copper and iron in an impure salt can do to your products?

These impurities form unappetizing scums and discolorations on vegetables. Slow down curing of meat, make it tough and bitter-tasting. Give frozen fish poor texture and fishy taste. Cause oxidative rancidity in foods containing fats and oils.

Today's food market is keen. Competitive. The quality of your product is one of your best sales weapons. Why spoil it with an impure salt?

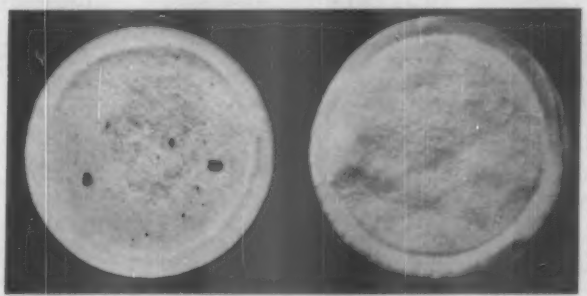
Why flake salt protects quality

Only Diamond Crystal is made by the exclusive Alberger system. This produces flake salt 99.95% pure. An amazing high purity that gives Diamond Crystal *true salt flavor* . . . without the bitter taste imparted by calcium and magnesium compounds, present in less pure salt. By using Diamond Crystal Flake you can *salt to the limit* for optimum flavor.

Diamond Crystal Flake averages less than 1.5 parts per million of copper or iron. Helps prevent oxidative rancidity and off-flavor development.

Are you using the right salt?

The grain size and rate of solubility of salt has an important effect on your product. That's why



Unretouched photograph of actual filter test shows difference in purity and cleanliness between a leading granular, cube-like salt (left); and Diamond Crystal Flake Salt (right).

Notice how impurities in the granular salt used discolored the corn on the left—while pure Diamond Crystal has protected the golden appeal of the corn at right. Which brand do *you* think the housewife would buy again?

Diamond Crystal Flake Salt is precision screened into a wide range of grain sizes to meet the most exacting *individual* requirements of your product.

Send for free analysis

If you are not sure that the salt you're using is suitable for your product, send a sample of it with the coupon below to Diamond Crystal. The salt will be given a careful laboratory test and the results forwarded to you free of charge.

Mail this coupon for free salt analysis

Technical Department FT
Diamond Crystal Salt Co., St. Clair, Michigan

I enclose a sample of the salt I now use. Please have it tested and send me the report free of charge. I would also like information on special Diamond Crystal Salts made for my industry.

Name_____

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Product_____

Address_____

City_____ State_____

• LOCATION of enamel break-through, peppering and exposure is SEEN instantly. Just reverse current and look for the bubbles.

• AUTOMATIC TIMING helps avoid serious reading errors. Red light turns on when electrode is immersed. Red turns off and green light turns on a few seconds later when it is time to read MA.

• SUBMERGED LAMP avoids glare, perfectly illuminates INSIDE of can to show bubble formation.

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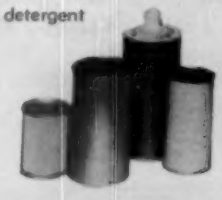
Developed with the help of and used by the major can makers of the U. S. The Enamel Rater is one of several can inspection tools thus produced by WACO for quality control in the packing and canning industry!

• ALL SIZES of detergent and beer cans.



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Laboratory Supplies and Equipment
WILKENS-ANDERSON CO.
4525 W. Division St. Chicago 51, Ill.

(Continued from page 44)

STERLING, C. (Univ. of California, Davis). *Food Research, 21,* 546-54 (1956).

When time of loading is varied, simple mechanical models are inadequate for the interpretation of rheological behavior of starch gels. Crystallization, or formation of new micellar bonds, seems to account in part for the departure from idealized models. Displacement of isolated and lightly attached polysaccharide mols. ("brush heap") and breakage of weaker bonds are probably involved also. Increased stress time gives an increase in time of strain retardation and in permanent deformation and a decrease in resilience. With repeated loadings the gel shows a higher elastic modulus and decreased permanent deformation. "Work-hardening" is thus demonstrated.

Triose compounds in maple sirup.

UNDERWOOD, J. C., LENTO, H. G., AND WILLITS, C. O. (Eastern Regional Lab., Philadelphia 18, Pa.). *Food Research, 21,* 589-97 (1956).

Presence of triose, probable precursors of maple flavor and color, has been demonstrated in the steam distillate of maple sirup. The major portion of these compds. is probably acetol.

System for classification of structurally related carbohydrates.

ISBELL, H. S. *J. Research NBS, 57,* 171-8 (1956).

A system is presented for the classification of structurally and configurationally related carbohydrates. Each substance is assigned a code no. that defines the structure and configuration. By inspection of the code nos., or by a punched-card technique, groups of structurally related carbohydrate derivs. can be selected readily from a heterogeneous collection. The structures, configurations, and conformations of pyranose and furanose derivs. are discussed, and classifications are made on the basis of a few fundamental structures.

TASTE AND TASTE TESTING

Group size in taste sorting trials.

GRIDGEMAN, N. T. (Natl. Research Council, Ottawa, Canada). *Food Research, 21,* 534-9 (1956).

Experimental trials indicate that small flavor differences are more efficiently detected when the coded samples are presented as 4N pair comparisons than as N octads to be sorted by taste. The statistical theory of such trials is discussed. The tentative generalization is made that the larger the group presented to the subject in a taste trial the less efficiently can he exercise his ability to discriminate.

INDUSTRIAL HEALTH

Studies of ozone toxicity. I. Potentiating effects of exercise and tolerance development.

STOKINGER, H. E., WAGNER, W. D., AND WRIGHT, P. G. (Public Health Service, U. S. Dept. of Health, Education and Welfare). *Arch. Ind. Health, 14,* 158-62 (1956).

A striking enhancement of the toxicity of ozone was demonstrated in rats and mice when they are exercised intermittently during exposure. Ozone concns. of 1 p.p.m. are fatal in 6 hrs. in these species when accompanied by exercise for 15 mins. each hr. during exposure. A marked tolerance to ozone was developed in these species.

ORGANIC CHEMISTRY

The acetylation of organic hydroxy compounds with ketone.

DUNBAR, R. E., AND BOLSTAD, L. L. (Minneapolis-Honeywell Regulator Co., Minnesota). *J. Org. Chem., 21,* 1041-4 (1956).

The use of ketene for acetylating of glycols (including carbohydrates) was found practical. Ketene, prepd. by pyrolysis of Me_2CO used in CCl_4 solvent, gave essentially quant. yields. A few drops of 18*M* H_2SO_4 served as catalyst.

The enzymic synthesis and degradation of starch. Part XXII. Evidence of multiple branching in waxy-maize starch. A correction.

PEAT, S., WHELAN, W. J., AND THOMAS, G. J. (Univ. Coll. of North Wales, Bangor). *J.Chem. Soc.* (1956) 3025-30.

Additional evidence confirms that the mol. of the β-limit dextrin, and hence that of the parent amylopectin, is multiply branched on the random pattern postulated by K. H. Meyer.

PHYSICAL RESEARCH

Finely divided solids as emulsifiers.

MUKERJEE, L. N., AND SRIVASTAVA, S. N. (Lucknow Univ., Lucknow, India). *Kolloid Z., 149,* 35-8 (1956).

Basic salts form emulsions considerably coarser but quite stable. The basic carbonate systems were very much inferior to basic sulfates.

The solubility of tristearin in organic solvents.

HOERR, C. W., AND HARWOOD, H. J. (Armour and Co., Chicago). *J. Phys. Chem., 60,* 1265-9 (1956).

The soly. of tristearin was detd. quantitatively in hexane, benzene, CCl_4, $CHCl_3$, Et acetate, acetone and ordinary unhydrogenated cottonseed oil. The soly. of this triglyceride is decreased markedly by increased polarity of the solvents. In the presence of solvents, tristearin exhibits three distinct soly. curves corresponding to the three cryst. forms established previously by m.p. detns. and x-ray measurements. The occurrence of a fourth cryst. form reportedly melting at 70° is not confirmed by the soly. studies.

RADIOCHEMICAL TECHNOLOGY

Radiation—helpful or harmful?

ZAVON, M. R. (Univ. of Cincinnati). *J. Am. Med. Assoc., 162,* 532-5 (1956).

The author discusses the medical use and misuse of ionizing radiation and safe conditions surrounding its use. He advocates 1) formal training in radiation therapy for all physicians who will use x-ray or Ra, 2) application of the newer knowledge of radiology in their practice.

REGIONAL SECTIONS

(Continued from page 30 preceding technical papers)

diarrhea, and a very high death rate among children in the age range 1 to 4. In its severe form, the disease is generally called "kwashiorkor," or in Latin America, "syndrome pluricarencial infantile." Although the major deficiency in this characteristic disease is a lack of good quality protein, concurrent deficiencies of vitamin A and riboflavin are very common, and deficient intakes of calcium, vitamin C, iron and fats are not uncommon. The degree to which other deficiencies are involved varies from section to section and can be estimated on the basis of dietary studies and clinical surveys. The death rate, stunting of growth, anemias, edema (swelling), fatty livers, pancreatic injury, atrophy of the intestines, and general impairment of health within the pre-school age group, make the problem of education and direct assistance extremely difficult. The intake of animal protein foods in these countries may run as low as 5% of the total calories.

Dr. John Brock and his associates in South Africa have shown that a mixture of pure amino acids, including the eight shown by D. W. C. Rose (University of Illinois) to be essential in human nutrition, can cause at least an initial stage of cure, in treating kwashiorkor. As a public health measure, this approach is still impractical. Complete restoration of health would not occur without furnishing all of the nutrients that are required in a balanced diet. In practical terms, this means working toward diets that include, in addition to cereals, green leafy foods, fruits, legumes selected on the basis of their available amino acids, and the use of animal protein foods in so far as they can be made available.

Physicians are in complete agreement in their experience that the disease, kwashiorkor, if it has not progressed too far, is usually cured in a practical sense by an adequate intake of skim milk, meat, fish, or eggs. These foods also furnish minerals and a generous source of vitamins in the B-complex, in addition to calories and flavor. Low-cost fruits and vegetables are grown in nearly all major tropical areas, and could furnish a generous source of vitamins A and C in addition to minerals, sugars and proteins. Their adequate use depends, however, on education—and lack of nutrition education is almost as serious in many of the areas as a lack of working capital or fertile land. It is common practice to distribute vitamin A capsules with non-fat milk powder in the medical clinics, but a satisfactory way to furnish stabilized vitamins A and D in the non-fat milk powder apparently has not been developed. The problem merits careful study.

Research on Plant Proteins Merits Support

In India where vegetarianism (except for milk acceptance) is a common practice, research on the plant proteins has been relatively active, as it has more recently in Central America, Africa, and elsewhere. This kind of study merits world-wide coordinated support. Sesame seed and cottonseed, for example, contain protein of good quality and can be grown in most of the areas where deficiencies are severe. Cottonseed requires special processing, however, to remove gossypol. Some of the Indian legumes (grams) also show considerable promise as single or blended protein foods. Wheat, corn, rice, oats, beans, peanuts, soybeans, and cocoanut presscake can make an important contribution toward complete protein sources if used properly with other foods. The rice and fish diets of the Orient are famous for their efficiency. In the countries where kwashiorkor still persists, the foods required for a good diet could be developed and introduced rapidly if supported by a modest amount of education, research, and working capital. The booster sources of protein should be selected with care, but the added quantities need not be large. There is a critical need for testing the potential low-cost foods that would prevent protein malnutrition. The tests should include sequential studies with experimental animals, normal and protein-deficient children (under careful observation), and finally representative population groups to assure satisfactory performance in practice.

Very recently, the Rockefeller Foundation furnished a special fund to the National Research Council to accelerate research work on an international basis. The United Nations agencies (UNICEF, FAO and WHO) are working in cooperation with the above groups and with many private foundations and government groups in a world-wide attack on the central problem of protein malnutrition.

A Needless Sacrifice

The present needless sacrifice in human life and health literally includes millions of persons in the technologically less advanced portions of the world. During the weaning and post-weaning period the death rate often reaches the shocking range of 10 or more times higher than in the United States and Western Europe. There is no adequate basis for appraisal of the injury inflicted on those who survive. Physical stunting is often equivalent to 2 or 3 years of growth, and there are further penalties evident in depressed tissue functions and altered structures. Progress is going to be slow and difficult in working toward nutritional goals such as we enjoy, but that is all the more reason for tackling the job. Increased milk, meat, fish, and poultry production is getting under way in many of the countries where little progress has been made for centuries, but the necessary educational support is barely beginning. Increased use of plant foods, trace elements for livestock, antibiotics, silage and non-protein nitrogen feeding, vaccines, modern sanitation, and food engineering all have an important part to play.

Better Protein Nutrition, Better Neighbors

You ask "why has this condition persisted through decades and centuries?" A primary cause has been the failure to recognize until recently the nature and seriousness of the problem. This lag in defining the problem was especially true with respect to the feeding of very small children. Equally critical was the need for trained personnel in each part of the world, to guide research and education in the basic and applied aspects of agri-

culture and human nutrition. Perhaps I should add that our best nutrition scientists from the United States, Canada, Europe and other favored parts of the world are constantly adding to their own knowledge, too, as they work with local scientists in areas where protein malnutrition—frequently rated as "health problem number one"—is still severe. As the problems are solved, both they and we will be better neighbors.

PRESIDENT GARNATZ MAINTAINS FULL SPEAKING SCHEDULE

With eight regional section speaking engagements behind him and five ahead George Garnatz, IFT President, will soon have sampled the climate and characteristic banquet fare of most of the geographical regions of the U.S.A. Ahead lie the New England region (Northeast Section, March 7), the Midwest (Ames, March 15), the South (Florida, March 21), the North Central (Wisconsin, March 29) and the East (New York, April 17). At Madison, Wisconsin on March 29, Mr. Garnatz will present the charter at the induction ceremonies that bring the newly founded Wisconsin Section officially into the IFT fold.

In months past he has had an even more diversified schedule. In November, 1956, President Garnatz spoke to the Philadelphia Section on the 13th and to the Maryland Section on the 14th. On December 3 he was guest speaker at the Western New York Section meeting in Buffalo and on December 13 at the meeting of the Texas Section. During January he spoke four times —before the Indiana Section on the 12th, the Chicago Section on the 14th, the Great Lakes Section on the 25th, and the Ak-Sar-Ben Section (Nebraska) on the 30th. The Far West Sections were covered earlier in his term and were also served by President-Elect Emil Mrak of the University of California.

In addition to presenting his message to the regional sections, Mr. Garnatz held informal conferences with regional officers in order to learn their views of IFT plans and policies. The State of IFT over the nation is good and a healthy growth is apparent everywhere.

PHILADELPHIA . . .

The annual Ladies' Night was held on December 5, 1956, at Cavanaugh's Restaurant. One hundred and five members and guests were in attendance.

Dr. David W. Anderson, Director of Research of the Prescription Product Division of the Borden Co. was the speaker of the evening. The topic "Allergic Foods of Interest to the Food Technologist" was informative and interesting to the specialized food technologists as well as to the lady guests.

It is estimated there are 15,000,000 people in this country who are in need of some help for allergic conditions. Some of these conditions are attributed to the intake of some particular food. In addition 25% of all children have some degree of allergy and at least 7% of the children are allergic to milk. Other foods such as eggs, wheat, chocolate, nuts, cheese, fish and shellfish have been incriminated. With this situation it becomes understandable why it is a problem that should interest the food technologist.

Dr. Anderson stated that despite the fact that much is known of the types of foods that produce these allergic conditions there is a need for much fundamental work to understand the specific ingredients which are responsible.

Interest at this meeting was indicated by the many questions asked by the guests and at the conclusion all members and guests went home with a supermarket bag full of delightful gifts, groceries and "goodies" contributed by the member firms.

DIXIE . . .

The first winter meeting of the Dixie Section was held on 14 December 1956 with 22 members and visitors touring the bottling plant of the Atlanta Pepsi-Cola Co., a modern facility located in a new industrial development area on Chattahoochee Ave., Atlanta. In the evening, dinner and a brief business session conducted by President T. W. Kethley were held in the Alumni Memorial Building at Emory University with 33 members and guests present. Mr. K. B. McKenzie, in charge of quality control in the Southeast for the Pepsi-Cola Company, spoke on "Production Control in a Soft Drink Industry" with emphasis on methods being practiced by the Pepsi-Cola people. Mr. A. S. Perry, Section Program Chairman, completed the program with the showing of two excellent films on the story of chocolate.

A source of deep regret to all members was the announcement of the resignation of our faithful secretary-treasurer for the past five years, Mr. Carrol Neff. Mr. Neff has accepted a position with Foremost Dairies, Inc., in San Francisco, heading their nutrition research program. Mr. Neff along with his entire family has contributed greatly to the life and growth of this section and we certainly hate to lose him to the Northern California Section. Congratulations Northern California!

William B. Cown, Engineering Experiment Station, Georgia Tech, was appointed to fill Mr. Neff's unexpired term.

SOUTHERN CALIFORNIA . . .

The names of the new officers of the Southern California Section were annuonced at the December meeting of the Section. These are:

Chairman—E. B. Oberg, Carnation Research Center
Chairman-Elect—C. Gordon Beisel, Sunkist Growers
Secretary—Gordon Miller, C. & H. Sugar
Directors—Rodger T. Reid, Braun Corp.; Henry M. Espoy, Barnett Laboratories, Inc.

The December meeting, which is the annual Ladies' Night, was addressed by Dr. Emil M. Mrak, President-Elect of the Institute. His observations on the food industry in Brazil and Switzerland proved both pene-

trating and interesting to the 85 members and their guests.

NORTHERN CALIFORNIA . . .

Let's get up-to-date: Professor Eugene L. Grant, Professor of Economics of Engineering, Dept. of Civil Engineering, School of Engineering, Stanford University, addressed the Section on October 18, when his subject was "Statistical Quality Control." . . . The Section's 2nd Golf Tournament was held at the Richmond Golf Club, Richmond, Calif., in conjunction with the October 18 meeting, the latter, by the way, taking place at the El Jardin Restaurant, San Francisco. . . . On November 13 the Section and the Nutrition Foundation held a symposium on basic nutritional research and related aspects of food technology. Held at the Claremont Hotel, Berkeley, the symposium started with a luncheon at which Mr. H. O. Banks, Director of the California State Department of Water Resources, spoke on "The Outlook for Water Resources in California." During the afternoon Professor Daniel I. Arnon, Professor H. O. L. Fischer of the University of California at Berkeley, Professor Max Kleiber of the University of California at Davis, Dr. Wendell Griffiths and Dr. C. G. King of the Nutrition Foundation, delivered addresses in their respective specialties. It concluded with a dinner at which Dr. W. M. Stanley, Director of the Virus Laboratory, spoke on "Present Concepts of Viruses and Their Role." . . . IFT President George Garnatz met with the Executive Committee on October 11 and spoke informally about activities of national IFT. . . . The Annual Meeting was held on December 6 at the Western Utilization Research Laboratory, Albany, Calif., program for which was as follows:

MORNING SESSION

M. A. JOSLYN, Chairman

9:00- 9:30—Registration

9:30- 9:50—How Food Processors May Get Maximum Value from Their Technologists
J. L. Heid, Editor
Western Canner and Packer
San Francisco, Calif.

9:55-10:15—The Diversified Role of Food Technology in the Development of the Citrus Products Industry
L. C. Gallagher, Manager
Industrial Sales Division, Products Department
Sunkist Growers, Inc.
Ontario, Calif.

10:20-10:40—Ten Weeks in a Cannery
B. Feinberg
Fairview Packing Co.
Hollister, Calif.

10:45-11:05—Effect of Water Volume and Methods of Application on Tomato Washing
W. A. Mercer and W. Rose
Western Research Laboratory
National Canners Association
Berkeley, Calif.

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Sales Engineers in Principal Cities of U. S. and Canada

11:10-11:30—Measurement of Salt Penetration in Raw Tuna
E. Benjamini, P. Cambour and F. C. Lamb
Western Research Laboratory
National Canners Association
Berkeley, Calif.

11:35-11:55—Some Chemical Engineering Aspects of Research on Food Process Development
W. D. Ramage
Western Utilization Research Branch, U. S. D. A.
Albany, Calif.

12:00-12:10—Business Meeting

12:10- 1:10—Lunch

AFTERNOON SESSION

Section A—H. Lineweaver, Chairman

1:10- 1:30—Problems Involved in Brandy Stillage Disposal in a Plant Designed for Municipal Waste Treatment
G. K. York II, M. A. Kelmick, D. Davis, R. H. Vaughn
Dept of Food Technology
University of California, Davis, Calif.

1:35- 1:55—Volatile Aroma Materials from the Muscat of Alexandria Grape
D. Webb and R. E. Kepner
Dept of Viticulture & Enology and Dept of Chemistry
University of California, Davis, Calif.

2:00- 2:20—Yeasts Associated with Drosophila in Tomato Fields of Northern California
R. de Camargo and H. J. Phaff
Dept. of Food Technology
University of California, Davis, Calif.

2:25- 2:45—Volatile Reducing Substances in Canned Freestone Peaches
B. S. Luh, S. Leonard and L. L. Claypool
Dept. of Food Technology & Dept. of Pomology
University of California, Davis, Calif.

2:50- 3:10—Identification of Some Volatile Flavoring Constituents of Strawberries
K. P. Dimmick and J. Corse
Western Utilization Research Branch, U. S. D. A.

3:15- 3:35—Test Paper for Detecting Peroxidase
H. J. Morris
Western Utilization Research Branch, U. S. D. A.

3:40- 4:00—Texture Changes in Frozen Red Sour Pie Cherries
M. Gee and R. M. McCready
Western Utilization Research Branch, U. S. D. A.

4:05- 4:25—The Lipoxidases of Peas, Beans and Alfalfa and Their Practical Significance
A. L. Tappel ad A. M. Siddiqui
Dept. of Food Technology
University of California, Davis, Calif.

4:30- 4:50—Factors Influencing the Suitability of White Potatoes for Processing
C. E. Hendel, W. O. Harrington and W. R. Mullins
Western Utilization Research Branch, U. S. D. A.

AFTERNOON SESSION

Section B—C. R. Havighorst, Chairman

1:10- 1:30—Tolerance of Frozen Poultry to a Wide Variety of Temperature Conditions
A. A. Klose and H. F. Pool
Western Utilization Research Branch, U. S. D. A.

1:35- 1:55—Chemical Changes Induced by Gamma-radiation of Meats, Poultry and Fish
A. L. Tappel, H. Groninger and F. W. Knapp
Dept. of Food Technology
University of California, Davis, Calif.

2:00- 2:45—Studies on the Mechanism of Direct Steam Injection Heating of Fluids
A. I. Morgan
Western Utilization Research Branch, U. S. D. A.

2:25- 2:45—Oxidative Deterioration of Fish and Fishery Products
D. W. Brown, E. Einset, A. W. Venolia, H. S. Olcott and A. L. Tappel
Dept. of Food Technology and Institute of Marine Resources
University of Calif., Davis and Berkeley

2:50- 3:10—Gas Chromatography Applied to Analysis of Amino Acids
I. R. Hunter
Western Utilization Research Branch, U. S. D. A.

3:15- 3:35—Oxidative Fat Rancidity in Walnuts and Other Nut Meats
A. L. Tappel, K. Urs and F. W. Knapp
Dept. of Food Technology
University of California, Davis, Calif.

3:40- 4:00—Quality Changes in Rough Rice During Storage as Affected by Moisture and Temperature
D. F. Houston
Western Utilization Research Branch, U. S. D. A.

4:05- 4:25—Continuous Laboratory-Scale Jelly Manufacturing
J. D. Ponting and D. W. Sanshuck
Western Utilization Research Branch, U. S. D. A.

4:30- 4:50—Effect of Maturity and Storage Temperature on Quality of Frozen Pears
W. C. Dietrich, F. E. Lindquist and

M. M. Boggs
Western Utilization Research Branch,
U. S. D. A.

DINNER MEETING

7:30—Some Experiences in Russia
Dr. K. F. Meyer, Director
Hooper Foundation for Medical Research
University of California Medical Center
San Francisco, Calif.

PITTSBURGH . . . .

The 31st official meeting of the Pittsburgh Section was held on November 13, 1956, at the Royal York Apartments Dining Room. The Executive Committee appointed a Constitution Committee to have the local Section incorporated. Dr. B. R. Suri, Chairman of Publicity Committee, gave the report on the publicity program. He suggested that each member regularly attending the meetings, should bring another member who is not in the habit of attending the meetings. And that each member should contact potential members of the food industry to arouse interest in our section.

Mr. William A. Scarlett of Minneapolis-Honeywell Regulator Co., Inc. was the guest speaker. His topic was "The Role of Instrumentation in the Food Industry." Mr. Scarlett discussed many of the quality aspects of food which can be recorded and controlled by instrumentation. They included texture, consistency, color and clarity, specific gravity and refractive index. All of these aspects can be recorded and the information can even be teletyped to a central location many miles away. Mr. Scarlett gave us an interesting prospectus. He predicted that instruments will eventually be able to record changes in flavor, and the nuclear emission will be used for analysis of butterfat in milk.

WESTERN NEW YORK . . . .

On December 3 an unusually interesting technical program was held in the afternoon, and a dinner meeting followed by a talk by the National President of the Institute of Food Technologists in the evening.

The entire meeting was held at the Peace Bridge Motel, Buffalo, N.Y. The program follows:

AFTERNOON TECHNICAL PROGRAM
2:00 P.M.

1. "Report on Studies Relating to the Quality of Freestone Peach Purees as Affected by Harvesting and Processing Conditions," by Dr. D. E. Wilson, New York State Agricultural Experiment Station, Geneva, N. Y.
2. "The Structure and Properties of Present Day Food Colors," by Dr. Samuel Zuckermann, Plant Manager, H. Kohnstamm & Co., Inc., Brooklyn, N. Y.
3. "Radioactive Isotope Tracer Techniques," by Dr. Gordon Harris, Chairman of Department of Chemistry, University of Buffalo, Buffalo, N. Y.

Open new markets with the help of EASTMAN antioxidants

Ever develop a food product which could be given only local distribution—or perhaps couldn't be marketed at all—because of limited stability? If so, it probably contained an oxygen-sensitive ingredient, such as an oil or a fat, which caused the trouble.

Let Eastman help you combat this problem with its complete line of Tenox antioxidants. Our staff of food technologists would welcome the opportunity of working with you on the proper use of these antioxidants. Assisting other food processors in this manner, we have helped send many fine products from the laboratory shelves to the market place.

Since Eastman manufactures the widest range of antioxidants, we have no axe to grind in favor of any one. You can be certain our recommendation will represent the most economical and effective stabilization possible against oxidative deterioration.

For more information about Tenox, write to Eastman Chemical Products, Inc., Kingsport, Tennessee—a subsidiary of Eastman Kodak Company.

Tenox Eastman food-grade antioxidants

DINNER MEETING

The evening speaker was Dr. George Garnatz of the Kroger Foundation, and National President of IFT. His topic: "Kroger Foundation—Consumer Research."

PERSONNEL

MR. CARROLL F. NEFF has been appointed to head the nutrition research program of Foremost Dairies, Inc., San Francisco, California.

DR. D. E. BRADY, University of Missouri, Columbia, is on leave from his post to undertake an assignment with the Livestock and Meat Products Division, Foreign Agricultural Service, U. S. Department of Agriculture, Washington 25, D. C.

UNIVERSITY OF MASSACHUSETTS CHAPTER OF PHI TAU SIGMA ELECTS NEW OFFICERS AND MEMBERS

The University of Massachusetts Chapter of Phi Tau Sigma, the food science honor society, elected its officers for 1957. New President will be Dr. W. B. Esselen (Food Technology Dept.); Vice-president, Dr. R. B. Read, Jr. (Bacteriology Dept.); Secretary, Prof. B. Gersten (Experiment Station); Treasurer, Prof. Dorothy Davis (School of Home Economics); National Councilors, Professors K. M. Hayes, and A. S. Levine (Food Technology Dept.).

Seventeen new members were also elected to the Society. The group includes two U. of Mass. staff members, Professor N. G. Cournoyer (Food Management) and D. A. Evans (Dairy Dept.), two outstanding undergraduates, Miss Carcella Barca (Food Technology) and Phillip R. Wells (Dairy), and three alumni, M. S. Abdel-Rahman, M. A. Afifi and A. Sedky. Others elected to membership were Miss Georgia P. French, graduate student in Nutrition, A. Bolaffi, L. R. Fare, M. Gersham and N. L. Norcross, graduate students in Bacteriology and Public Health, and Wm. Draper, M. A. Jimenez, J. D. Shore, and E. Yanez-Cortes, graduate students in Food Technology. Professor Herbert W. Miles of Wye College (University of London) was also elected.

FOOD INDUSTRY TOPICS . . .

. . . by Norb Leinen

. . . in the public interest

Improved packaging of food products is the principal factor in achieving significant cost reductions in finished consumer goods, according to John A. Warren, technical advisor to the Packaging Division of American Management Assn., who addressed the New York Chapter of IFT on November 21. Pointing to the trend to packaging innovations, he cited as examples the combining into a single package of a cake mix, icing mix, and cake pan, as well as the creation of multiple unit packages by use of sleeves, and prepackaging of fresh produce. He noted that what often appear to be new products are frequently established items in new packages.

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An increasing variety of spread-type foods in fold-up metal tubes appears slated for grocery shelves throughout the U. S. in 1957, according to Lester B. Platt, executive secretary of the Collapsible Tube Manufacturers Council, in a year-end review of industry production. A Pennsylvania food firm is preparing to test-market peanut butter in a metal tube. A New York concern now packaging a jelly-like coffee extract in tubes as a flavoring for wholesale bakers and confectioners is expected to market a retail version of the extract. In Maryland, research is underway in tube-packaging of fish products. Metal tubes for food products account for only 1% of annual production that now exceeds one billion units. Shipments in this end-use category in the first 9 months, Platt reported, are about 10% ahead of the same 1955 period.

Announcements have been mailed, inviting applicants to send in their qualifications for the 1957 fellowship in the field of food refrigeration. The 1956 winner was Lawrence Spiegel of the Department of Food Technology, MIT. Address The Refrigeration Research Foundation, 12 N. Meade Ave., Colorado Springs, Colo.

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Radishes subject to a storage and transit disease known as "radish pit" were 50% less susceptible when dipped in an oxytetracycline solution, according to Dr. R. S. Cox of the University of Florida, Everglades Experiment Station, who spoke before the American Phytopathological Society in Cincinnati on December 7. According to Dr. Cox, symptoms of this disease, a serious threat to Florida's radish industry, appear initially as water-soaked areas within 48 hours after harvest. These areas develop into pitted lesions within 3 to 5 days. Oxytetracycline, however, gave striking control over this disease, and was found to be much more effective than streptomycin, which also was tested.

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An adequate total supply of food for this country and substantial quantities available for export was the prediction made by Paul S. Willis, recently re-elected president of the Grocery Manufacturers of America, at the association's 48th annual meeting in Philadelphia. This should mean, too, that food prices will continue fairly stable at present levels, which are 3% below what they were in 1952 when food prices were at their peak. He also pointed out that average

weekly earnings of manufacturing employees since 1952 have risen from $67.97 to an estimated $80.

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A 3-year research program designed to advance scientific understanding of the physical nature of tenderness in meat and to provide an insight into the changes induced by various tenderizing substances and agents has been undertaken by the American Meat Institute Foundation at the University of Chicago, 939 E. 57th St., Chicago 37, Ill. The program was activated on September 1 and is under the joint direction of Dr. Harry Wang, chief of the division of histology, and Dr. C. Edith Weir, chief of home economics. The new program actually is a broadening and extension of work previously accomplished in the same direction. As was the earlier research, the program in part is financed by joint grants from Huron Milling Co., Harbor Beach, Mich., and Paul Lewis Laboratories, Milwaukee. This study is in addition to and distinct from another meat tenderization study on frozen-dried meat already in progress at the Foundation pursuant to a contract with the Quartermaster Food and Container Institute for the Armed Forces. It is anticipated that the new investigation will furnish information useful for devising improved or alternative tenderizing procedures for commercial application.

. . . things new under the sun

Citrus beverages with a cleaner fruit flavor and more stable cloud can be achieved with the new DENSITOL® (Brominated Sesame Oil, Abbott), according to announcement from the Chemical Sales Division of Abbott Laboratories, North Chicago, Ill. The original DENSITOL, introduced in 1946, was brominated apricot kernel oil. The search for a new brominated oil was prompted by the problem of obtaining adequate supplies of apricot kernel oil. The name DENSITOL was retained because the new form will have essentially the same properties as the former oil, and no change in method of use will be necessary. Abbott also supplies two other brominated oils—GRAVINOL® and BROMINOL®—the latter the original brominated oil, introduced by the firm over 25 years ago.

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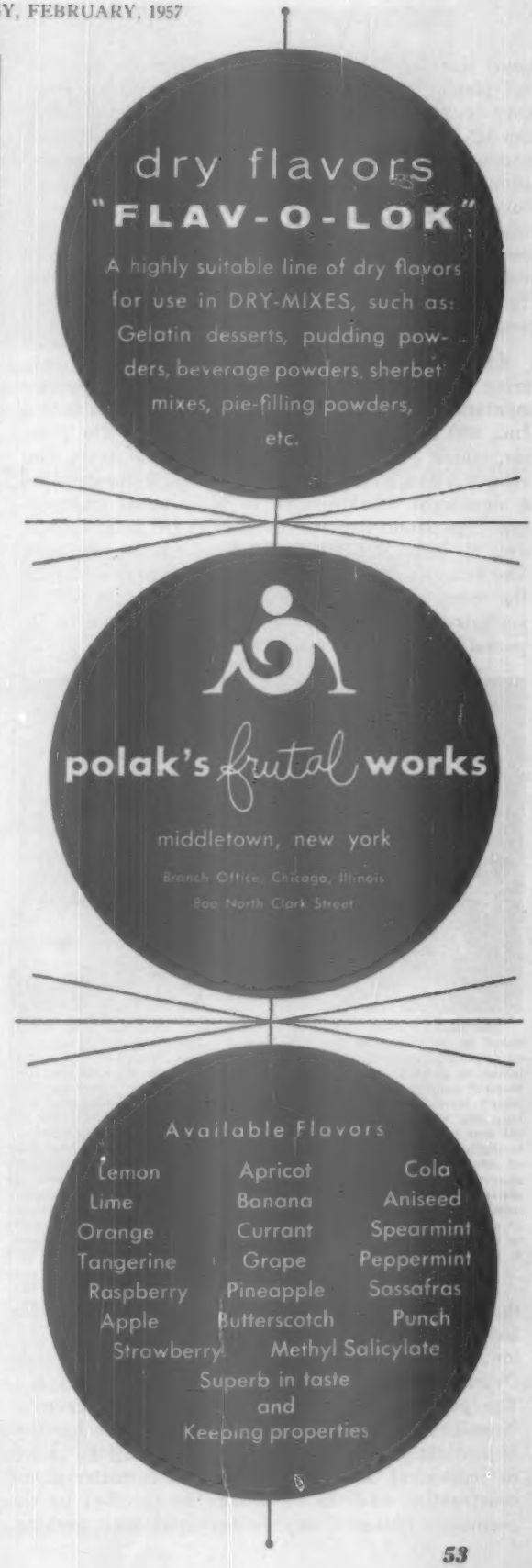
A new pressure can, an improved "Non-Drip" container, and a full line of detergent style and oblong, or I-style, cans have been introduced by American Can Company. Canco's new "Queen Size" 202 x 509 8-ounce pressure can, a complement to the 16-ounce "King Size," is intended especially for cosmetic-type products and can be furnished with either a 1" cup-opening top or the simple one-piece top. The new "Accu-Por" can, in 16- and 32-ounce sizes, is an advance in Non-Drip containers for liquid products. The one-piece formed top incorporates a non-drip nozzle to cut off flow of liquid after pouring. Either an economical metal screw cap or an Alseco aluminum cap can be employed with the container. The line of detergent style cans includes 12-, 16-, 22-, and 32-ounce sizes. All can be fitted with plastic nozzles and plastic screw caps, and the pint and



quart sizes can be furnished with either the standard ¾" plastic nozzle or ½" plastic nozzle. Canco's oblong or I-style can, stated to be the first solderless can of its type on the market features a clinched nozzle that prevents corrosion and flux splashes and allows a lithographed top, a non-drip spout with an outward curl on the neck that eliminates raw inside edges, compact stackability (made possible by a small recess in center bottom of can), and cemented sideseam, which permits lithography all around the can. The combination of cemented sideseam and clinched nozzle permits complete inside enamel.

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Exclusive rights to U. S. Patent No. 2,771,396, covering **a process for manufacturing L-Lysine by fermentation,** have been acquired by Chas. Pfizer & Co., Inc., 630 Flushing Ave., Brooklyn 6, N.Y. The process, which produces one of the 8 amino acids considered essential to human nutrition, is regarded as a significant breakthrough in biochemical engineering. The patent lists the inventor as Dr. L. E. Casida, Jr., of Pfizer's Biochemical Research Laboratories. The firm announces that L-Lysine Monohydrochloride, the form in which the essential amino acid will be marketed, will be priced from $12 per pound in 50-pound lots to $14 per pound in one-pound lots.

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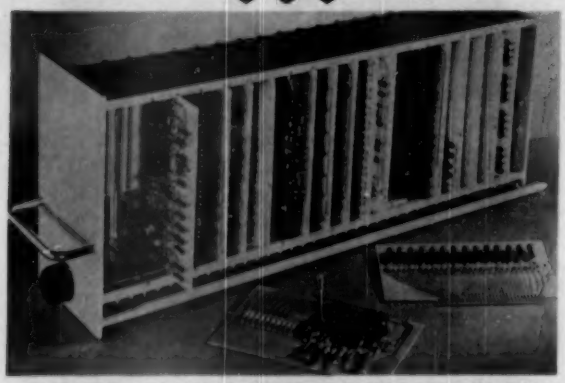

The new Beckman "112" Data Processing System has been introduced as the first such industrial system capable of continuously and automatically computing and logging secondary operating guides for industrial processes—often the controlling element in the safe and economical operation of a process. Instead of an expensive computer, simplified logic circuits, completely transistorized for dependability and long life, have been incorporated in the data system itself. Heart of the new instrument is the unique Beckman CADC (Computing Analog-to-Digital Converter). A single CADC is capable of handling hundreds of channels of incoming information from thermocouples, pressure gauges, stream-analysis instruments, etc. These data, in the form of electrical signals, are automatically sorted, stored, converted to digital numbers, or combined in complex mathematical formulas. The output in every case is in the form of simple numbers, easy to read, accurate to three figures, and ideally suited for production control. Manufacturer is Beckman Instruments, Inc., 2500 Fullerton Rd., Fullerton, Calif.

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A sanitary triplex pump with basic improvements that solve problems in the food and chemical fields has been designed by research engineers of the Votator Division of The Girdler Co., a Division of National Cylinder Gas Co., 224 E. Broadway, Louisville 1, Ky. The pump is employed where corrosive slurries of viscosities as high as 40,000 centipoises are handled at medium pressures. Standard design may be varied to individual service conditions. The materials of construction and the drive may be specified by the customer; plungers may be equipped with packing, cups, or "O" rings; a ceramic or special alloys may be used instead of the standard Stellite valve trim; flanged or sanitary connections may be employed, and higher pressures can be accommodated at reduced displacements. The 3 single acting cylinders are available with bores of ½", 2", 2⅜", or 2¾". Stroke is 2¾". Maximum capacities range from 10 to 30 gpm, and maximum pressures from 450 to 1500 psig.

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Two new additions to its family of ice-making equipment have been introduced by Cold Corporation of America, 1371 N. North Branch, Chicago. The CUBE-FREEZE Automatic Ice Cuber can be used as a companion unit for the CHIP-FREEZE (a 560-lb. automatic ice flaker), or as an individual unit. It produces a daily capacity output of 300 lbs. of fresh ice cubes, 1⅜" x 1⅜". The new ICE-CHIEF Automatic Ice Flaker produces 1,000 lbs. of fresh ice flakes per day. The new units are available in COCORA's range of decorator colors, as well as white and stainless steel.

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New Jak-Et-Pak multiple can packaging machine developed by Robert Gair Division of Continental Can Company, 100 E. 42nd St., New York 17, N. Y., packs crown-top or spout-top as well as flat topped cans in wrap-around boxboard carriers at speeds of up to 120 carriers a minute. Important in connection with the machine is a low-cost carrier carton which wraps around the cans from the top down and not only glues but also locks on the bottom. The combination of gluing and locking provides double protection against failure of the carrier and also eliminates need for the long compression section required for carriers that are glued only. The Jak-Et-Pak machine handles cans ranging in height from 2⅝" to 7¾", and in diameter from 2⅛" to 3⅝". These dimensions cover the full range of commonly used can sizes. The machine is expected to be made available in the future with provisions for packaging jars as well as cans. The versatile new system requires only one-third to one-half as much floor space as conventional systems.

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Completion of its **new line of flexographic printers designed specifically for high-speed printing on polyethylene** is announced by Lee Machinery Corp., 28 W. 23rd St., New York 10, N.Y. Available in from one to 4 colors, presses operate roll to roll set up to feed into folder, sheeter, or packaging machinery, and handle with equal facility and fine register paper, polyethylene, cellophane, foils, pliofilm, and cloth. The standard press is 27" wide, but is made available in width from 15" to 30". Printing repeats range from 4" to 35". Register may be adjusted laterally and horizontally while press is in operation.

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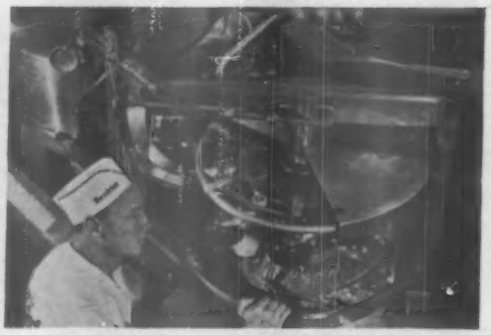
The new Duratite method of packing frozen, eviscerated poultry involves use of a new polyethylene film bag produced in 6 sizes and delivered plain or

(Continued on page 56)

One look at the huge, sprawling buildings at one of the Exchange Products plants and you know that big things are happening inside. For here *citrus* is king. Trucks pour in with fresh fruit from groves all over California and Arizona. Conveyer belts hum along in every direction. And Exchange products are shipped to nearly every part of the world.

Why 'Exchange' products serve you better



Utmost cleanliness is the watchword at Exchange Products plants. Everything has that "just washed-down" look. Every four hours, extraction equipment is completely cleaned. Every third day, the entire plant closes down for a thorough washing.

Exchange quality is assured by constant checking in extensive testing laboratories. In fact, rigid quality controls have been set up *throughout* the plant to make sure that every product measures up to Exchange quality. Sunkist produces more than 400 different citrus products.

The world's largest citrus research department is maintained by the Sunkist Growers to develop new growing methods, to improve manufacturing processes, and to perfect better products. Today, with a backlog of more than 60 years experience, Sunkist has accounted for 118 separate U.S. patents.

Just three of many Exchange products are *Exchange Preserver's Pectin*—guarantees a more uniform finished product; *Exchange Low Methoxyl Pectin*—jells *without* sugar, tasteless, controls "weeping" in pies; *Exchange Lemon Juice*—brings out natural flavor of *any* food. Superior acidulant, excellent anti-oxidant.

Sunkist Growers

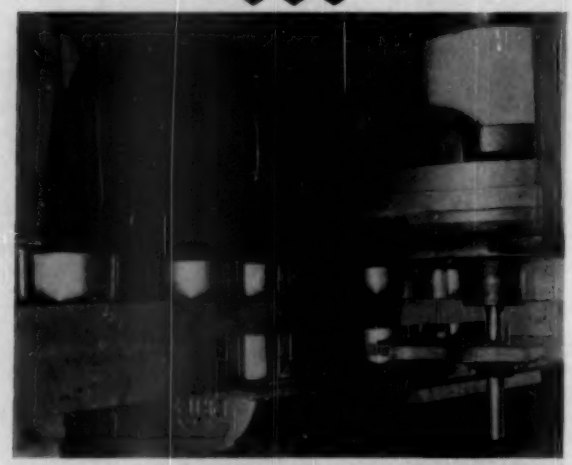
PRODUCTS DEPARTMENT • ONTARIO, CALIFORNIA

Distributors for

Exchange Lemon Products Co., Corona, Calif. • Exchange Orange Products Co., Ontario, Calif.

(Continued from page 54)

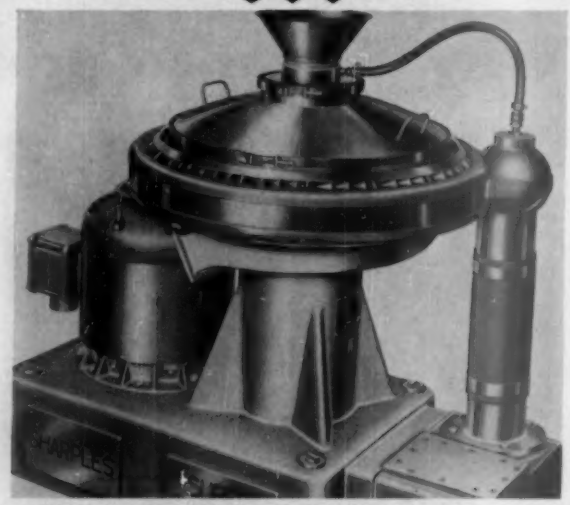
with the name and sales message on it, and a special Duratite machine for use by processors that replaces the vacuum tube and eliminates need for hot water bath or steam tunnel. The machine creates its form fit through use of grooved rollers, similar to the non-automatic washing machine wringers. The poultry acts as a piston in a cylinder, and the air is expelled as the bag forms and shapes the poultry into a compact, plump form. Advantages claimed are: 20% to 52% lower basic bag prices, depending on size; production line speed even with inexperienced operators; elimination of brittleness and bag shatttering, even at —90° F.; and pliability, which, despite a tight wrap, brings out the natural lines of the bird for eye-appeal. The Duratite machines can be purchased from Process Packagers, Inc., Minneapolis, at $450 f.o.b. Minneapolis. The Hydraphone clip fastener—the only one presently recommended for use with the process—can be purchased from Hydrahone Equipment Sales Co., Milwaukee, Wis. Price without air compressor is $395 f.o.b. Milwaukee. For details on the process, write Don Elder, vice president, Herb-Shelly, subsidiary of Brown & Bigelow, Farmington, Minn.



Close-up of Bulk Salt Precision Dispenser engineered by Diamond Crystal Salt Co., St. Clair, Mich., shows magnetic insert underneath conveyor belt which keeps can from falling off and brings empty cans to nozzle of Dispenser for uniform salt charge. Patented "live cone orifice" and heating elements in hopper base plate and dispensing plate keep salt dry and free flowing. Cans then move on to filling table. The machine dispenses at a rate up to a maximum of 180 cans per minute, and has been used effectively in canning tomatoes, green beans, beets, and asparagus. It reportedly is equally adaptable to dispensing both monosodium glutamate and calcium sulphate salt with mixtures. Manufacturer is Frank Hamachek Machine Co., Kewanee, Wis.

Aget Mfg. Co., Adrian, Mich., announces an addition to their line of Dust and Mist Collectors. The unit, the 90 NB 50, is designed for use in grinding, buffing, woodworking, and miscellaneous operations where a large column of dust, chips, shavings, and other residue accumulates from manufacturing processes. Special feature is the roll-away dust collecting bin, which has a storage capacity of 37½ cu. ft. The collector has an air handling capacity of approximately 10,000 CPM, and uses a 15 hp motor. The fan inlet will accommodate 15″ pipe.

An 8,000-lb. capacity model with dual drive wheels, the EUT-8024 is the newest addition to Clark Equipment Company's line of battery powered fork-lift trucks. A turning radius of 85″, aisle for right-hand stacking (including 48″ long load) of 148½″, and over-all length of 133″ are dimensional features. With 4 speeds forward and 4 reverse, it will travel loaded at 5½ mph and climb a 10% grade. The unit has 3 independent braking systems. An automatic "tilt-lock" valve in the hydraulic system prevents "drifting" of the uprights under heavy loads. Weight of the truck is 11,580 lbs. with lead acid battery, and 11,378 lbs. with alkaline battery. Write Electric Truck Section, Industrial Truck Division, Clark Equipment Co., Battle Creek, Mich.



The Sharples Super Classifier, the first dry powder classifier to successfully combine sharpness of separation and high efficiency with high capacity, operates on the air vortex principle. Available in 4 sizes with capacities ranging from approximately 1000 to 30,000 lbs. per hour, the compact unit provides an ultra-sharp cutpoint at high capacity regardless of feed rate or size distribution in the feed (fine fractions with a top size in the range of 15-20 microns will contain less than 0.01 to 0.04% 325-mesh screen residue). In addition, product recovery (yield) is considerably higher than by other means; exact percentages vary with density of powder, cutpoint desired, etc. Actual production runs on talc have been made with over 97% yield at a top size of 15 microns.

Two independent lubrication systems, providing both cyclic and continuous oil flow to different bearings on the same machine, are being built into a single automatic lubricator by Bijur Lubricating Corp., 151 W. Passaic St., Rochelle Park, N.J. Bijur's new AP-3 duplex lubricator is designed specifically for machines with a dual lubrication problem—where some bearings need periodic lubrication, others require oil continuously. Initially installed on a multiple spindle automatic bar machine, the AP-3 lubricator can solve dual lubrication requirements on many machine tools, textile machines, printing equipment, and special machinery, Bijur reports.

Designed to handle heavy-duty continuous grinding and other high dust volume sources, new Model 123 dust collector, the largest cabinet-type collector made by Torit Mfg. Co., 287 Walnut St., St. Paul, Minn., handles as many as 8 grinding wheels at a time. Dust storage capacity is 10 cu. ft., and filter

area is 300 sq. ft. A 5 hp motor moves approximately 2,000 cu. ft. of air per minute through an 8" inlet at speeds of more than a mile a minute. Cloth filters are chemically treated for spark resistance and sealed against leakage. Filters, motor, and blowers are easily reachable through removable doors. F.o.b. factory price is $1,198.

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The uniformly successful use of **wool felt as a thick dense filter medium with which to control air pollution,** collecting particles ranging in fineness from crushed brick dust to confectioner's sugar, is reported by American Felt Co., Glenville, Conn. The Hersey reverse-jet method of filtering dust and types of fume from air uses a thick dense filter medium—wool felt or similar structures made of synthetic fibers—to increase effectiveness of the filtration processes independent of the bridging of interstices such as exist in woven fabrics. End result, according to the company, is to permit filtering at a high velocity per unit area. The porosity of the filter and the constancy of the pressure drop are maintained by a slot-formed jet of very high velocity air injected through the medium in reverse direction to the filtration flow. The Hersey process was developed by H. J. Hersey, Jr., a mining engineer, in 1938. Mr. Hersey, whose offices are at 100 Park Ave., New York City, has four domestic licensees: The Day Co., Minneapolis; Koppers Co., Inc., Baltimore; Western Precipitation Corp., Los Angeles; and Pulverizing Machinery Division of Metals Disintegrating Co., Inc., Summit, N.J.

A new vacuum air purge device designed to remove air and objectionable odors from dairy products while improving efficiency of high-temperature short-time pasteurization has been developed by York Corp., subsidiary of Borg-Warner, York, Pa. The firm's engineers claim the new deodorizer reduces refrigeration requirements in the HTST system up to 20% and requires 30% less steam for the plant pasteurization process, thereby reducing operating costs. It provides fully automatic operation under all conditions without need for expensive code controls or attending manpower. The unit handles a variety of dairy products, including cream line milk, homogenized milk, vitamin "B" or "D" plain or homogenized, coffee cream, half-and-half cream, chocolate milk, and skim milk. Operating effectively as both a liquid and gas pump, the unit is said to considerably reduce homogenizer noise and pipe line vibration.

◆ ◆ ◆

Fire, which attacked the roof of a large new mushroom house, was snuffed out at the ceiling line by vermiculite insulation which had been poured over the ceiling. A product of Zonolite Co., 135 S. La Salle St., Chicago, Ill., Vermiculite is mined in Montana and So. Carolina and heat-treated in processing so that the granules expand as much as 12 to 15 times. In expanding, tiny dead air cells are entrapped in each granule, accounting for the mineral's extraordinary insulating value. Its fire-safety is a result of its high fusion point, in excess of 2200° F.

New "Built-in" Process Refractometer Assures Continuous... Accurate... Easy Quality Control

The internationally known GOLDBERG PROCESS REFRACTOMETER is designed for the continuous precise, rapid and easy determination of total soluble solids... in turbid and dark-colored concentrates, jellies, jams, pastes, purees, juices, syrups, preserves, milk products ... and in other solutions.

Three models are available in Brix ranges: 0 to 25%, 12 to 72% and 36 to 82%. Any model can be mounted on the side wall of a process vessel, evaporator, vacuum pan, fruit spread cooker, product pipe line or other location where continuous reading is desirable. The refractometer may also be mounted into a fixture that runs onto a track above a row of kettles for sample ladling.

The unique optical system transmits a sharp transition line from light to dark onto the large .5% graduated scale in the focusable eyepiece.

The illuminating unit consists of a 6 volt, ¼ ampere, long-life bulb ... detachable socket and cord.

The compact and rugged refractometer when mounted into the process vessel is vacuum and pressure-tight ... designed to withstand pressure of 20 pounds and temperatures ranging up to 110°C (230°F). The instrument is readily removable from coupling flange.

Instruments are factory calibrated. Recalibration to operating temperature is accomplished by a simple exterior set screw adjustment. A manually-operated wiper always brings fresh product sample to the prism face.

The GOLDBERG PROCESS REFRACTOMETER offers the following important advantages: accurate readings are instantaneous ... withdrawal of sample not necessary... evaporation errors eliminated... readings unaffected by viscosity, foam, air bubbles and pulp... no operator training period is required.

Why not write for further details today!

American Optical Company
Instrument Division, Buffalo 15, New York
Dept. N190
Gentlemen:

Send complete information on:

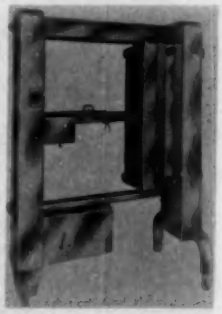
☐ Goldberg Process Refractometer
☐ Goldberg Combination Hand and Dipping Refractometer

Name _____
Address _____
City _____ Zone _____ State _____

Moeller Instrument Co., Richmond Hill 18, N. Y., again is offering without obligation its **temperature conversion chart** to engineers, students, and others having use for this pocket-size aid. Fahrenheit and Centigrade temperature equivalents are shown.

Prices on 316 stainless steel and Carpenter 20 alloy Series S Chempumps have been sharply reduced to start at $325—less than the cost of a conventional centrifugal pump of similar delivery characteristics, equipped with a mechanical seal. Chempumps feature leakfree operation, no need for lubrication, and simplified maintenance. Write Chempump Corp., Station P, 1300 E. Mermaid Lane, Philadelphia 18, Pa.

A new, all-stainless steel Plate Heat Exchanger featuring completely automatic push-button take-up and release control is announced by Kusel Dairy Equipment Co., Watertown, Wis. A fool-proof hydraulic system provides the smoothness and steady power to assure identically perfect, instantaneous take-up every time, even under higher-than-average pressures. The versatile new "DH" Series unit will accomplish any job from high-temperature short-time pasteurization to heating and cooling, and also offers the efficiency of regeneration wherever applicable. Its capacity can be increased or decreased to suit the need, or the entire unit can be shifted to a different job in the plant, assuring full production at all times.



Process-control engineers now have an entirely new and exceptionally dependable way to analyze industrial gas streams in the **first continuous gas chromatograph designed specifically for industrial processes.** The new Beckman Process Gas Chromatograph is a compact, thoroughly engineered device designed to make continuous samplings of any gaseous process stream. Each sample is passed automatically through a packed column of absorbent material which breaks the mixture into its component parts, and then registers on a graph the percent of each chemical component present. This information can be fed into a relatively simple control system which can keep track of the reaction and automatically correct any deviations. The first Beckman units, engineered and built by Watts Mfg. Co., Ronceverte, W. Va., already have rung up astonishing records of increased product quality and yield, even when used on existing processes. Beckman application engineers have been working to apply the instrument to a wide range of industrial processes in chemical manufacturing, petroleum refining, food processing, sanitation, and power. For further information write Beckman Instruments, Inc., 2500 Fullerton Rd., Fullerton, Calif.

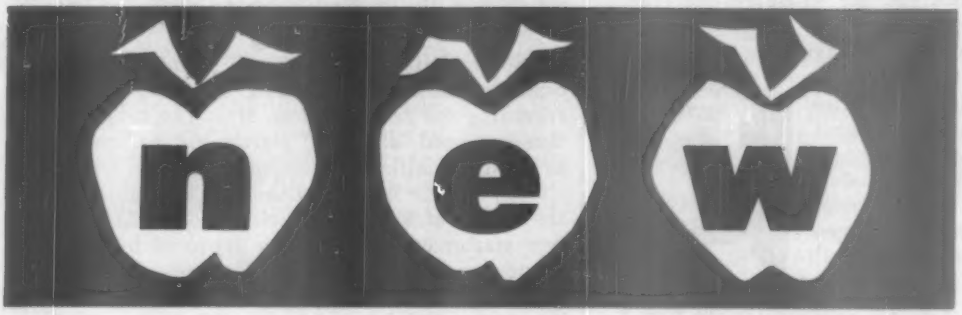
Two novel epoxy compounds, dipentene monoxide and alpha-pinene oxide, are available in experimental quantities from Becco Chemical Division of Food Machinery and Chemical Corp., Buffalo 7, N. Y. Both compounds are suggested for use as intermediates in the manufacture of perfumeries, flavorings, pharmaceuticals, protective coatings, insecticides, bactericides, lubricant additives, adhesives, plasticizers, and flotation agents; as reactants in organic synthesis, and as solvents.

Scientific testing equipment has been developed by food technologists of Food Research Laboratories, Inc., 48-14 33rd St., Long Island City, N. Y., to standardize testing of Flav-R Straws for quality control of flavor, sweetness, and color. The apparatus, an adaptation of the automatic intermittent pipette, has been dubbed "Sister Susie." Chief purpose of the equipment is to determine whether a youngster receives flavor and other qualities through a Flav-R Straw in equal quantities during the process of drinking a glass of milk. Flav-R Straws, in chocolate and strawberry flavors, contain a flavored pad, so that the milk sipped through them becomes chocolate- or strawberry-flavored. In the photo, Caroline Trent, nutritionist for Flav-R Straws, Inc., is shown adjusting the straw as one of the FRL chemists makes notes.

An improved silicone rubber for use in domestic and industrial oven door seals and in other "hot" applications has been added to the standard product line of the Silicone Products Dept., General Electric Co., Waterford, N. Y. Designated SE-751, this low compression set silicone rubber can withstand intermittent temperatures as high as 600° F. The product can be made harder by addition of certain diatomaceous fillers, has excellent processing characteristics for extrusion and calendering, and has low shrinkage and low water absorption. Properly fabricated SE-751 parts can qualify under AMS 3302 and ASTM TA BE, E_2F_2L.

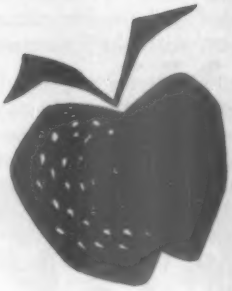
A new, completely automatic, entirely self-contained industrial Manostat, of special value in the precise control for distillation of sensitive materials, has been introduced by Manostat Corp., 20-26 N.

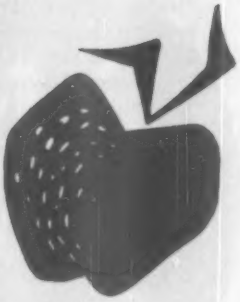
(Continued on page 60)



a better imitation

strawberry





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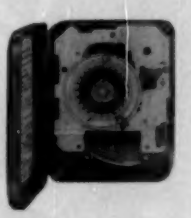
(Continued from page 58)

Moore St., New York 13, N. Y. The Cartesian Manostat No. 10, which allows users to achieve automatic vacuum and pressure control with a single instrument, is claimed to have almost human-like sensitivity and response. Outstanding feature is a permanent, accurate memory system. Once the vacuum or pressure is set in the Manostat, the setting will not be disturbed, even if the system is closed down and later reopened.

A new timer for controlling "on-off" cycles from 10 seconds to 12 minutes is offered by International Register Co., 2620 W. Washington Blvd., Chicago 6, Ill. Known as the Intermatic "Cycler-12" (Series C8301-S) timer, it is recommended for use on ventilating fans, pumps, process timings, and other industrial uses requiring flexible cycles of operation. Housed in a drawn steel case, the unit measures 6½" high by 5" wide by 3" deep. Presently, it is available only for 125v operation, with a capacity of 1875 watts. Write for Bulletin NR 156.



. . . *the literary corner*

Chromatographic Methods, a new publication to be issued several times annually, devoted exclusively to chromatography and the methods employed in chromatographic separation, is announced in the December 1956 issue of "Fatty Acid News Digest," published by Fatty Acid Producers' Council, 295 Madison Ave., New York 17, N.Y. Copies of *Chromatographic Methods* are available on request to: H. Reeve Angel & Co., Inc., 52 Duane St., New York 7, N.Y.

Three informative folders, "Profitable Packaging Ideas," released by Ekco-Alcoa Containers Inc., Wheeling, Ill., show new developments in packages for frozen meats, refrigerated meats, and frozen specialties. They feature suggestions on increasing sales by adding consumer convenience to product quality and by merchandising branded items with the attractiveness of rigid aluminum foil. Illustrations of many types of suggested packages for meat products are shown.

One-page Bulletin No. 427, offered by Proctor & Schwartz, Inc., 7th St. and Tabor Rd., Philadelphia 20, Pa., illustrates and describes the company's electrically heated laboratory dryer.

Bulletin No. 133 describes Niagara coil surface type air conditioners for industrial and commercial applications. Machines illustrated are offered in capacities from 41,500 to 1,680,000 BTU/hr. cooling range. Write to Niagara Blower Co., 405 Lexington Ave., New York 17, N. Y.

Control of dust from coal and coke handling operations is discussed in Bulletin 551-D, available from Wheelabrator Corp., 1095 S. Byrkit St., Mishawaka, Ind. Use of local exhaust ventilation and cloth-tube-type dust collectors is explained. Four illustrated case histories are presented.

Equipto, Division of Aurora Equipment Co., Aurora, Ill., announces availability without charge of its 48-page 1957 Reference Manual of Steel Equipment No. 485.

A reprint of a paper entitled "Apparatus and Method for the Determination of the Volatile Oil Content of Spices," which appeared in the August 1956 *Journal of the Association of Official Agricultural Chemists,* is available without charge by writing the *Gentry Serenader,* Gentry, P. O. Box 2076, Terminal Annex, Los Angeles 54, Calif.

"Seven Steps to Controlled Production of Tangy Summer Sausage," providing detailed information regarding the new American Meat Institute Foundation-developed lactic acid starter culture, specifically as used to initiate development of the traditional "tangy" flavor in summer sausage, is obtainable from the AMIF at 939 E. 57th St., Chicago 37, Ill. The new starter culture has been approved by the Meat Inspection Board for use in Federally inspected establishments "in the preparation of such kinds of sausage as thuringer, lebanon bologna, cervelat, salami, pork roll, and summer sausage." Large volume production facilities now are being converted for production of the culture by Merck & Co., Inc., licensed to prepare and distribute the culture under a non-exclusive agreement with the Foundation.

The new 12-page 1957 Reference Guide to Dow Corning Silicones describes almost 150 commercially available silicone products, including several 1956 developments. Products again are grouped by usage (water repellents, dielectrics, release agents, etc.), enabing engineers to locate a material by what it does as well as by what it is. Heavily illustrated with application photographs. Write Dow Corning Corp., Midland, Mich.

Reprints of articles, "Permeability of Acetostearin Products to Carbon Dioxide, Oxygen, and Nitrogen," and "Permeability of Acetostearin Products to Water Vapor," published in *Agricultural and Food Chemistry* in July and May 1956, respectively, may be obtained without cost from Southern Utilization Research Branch, USDA, 1100 Robert E. Lee Blvd., New Orleans 19, La.

Catalog 2, 32 pages, describing the company's products available for "immediate shipment," is offered by Fischer & Porter Co., 907 Jacksonville Rd., Hatboro, Pa., manufacturer of complete process instrumentation. Also available from F&P are Catalog 70-27, describing the company's rotating vacuum transmitter for automatic chlorine proportioning, and Catalog 70-21, describing the firm's chlorine evaporator.

A 32-page report designed to help with cost estimation and selection of impervious graphite processing equipment and covering all the standard impervious graphite processing equipment produced by Falls Industries, Inc., Aurora Rd., Solon, Ohio, under the brand name "Impervite," covers heat exchangers

of the tube and shell, cubical, cross-bore, plate, and bayonet types, as well as cascade coolers, HCL absorbers, towers, centrifugal pumps, rupture disks, pipe, fittings, and valves. A relatively comprehensive chart of chemical resistances is given, together with a brief outline of properties and characteristics of Impervite impervious graphite, where is is used as a material of construction for processing equipment. Write on company letterhead to Mr. Geo. Sarvardi.

A newly revised 16-page edition of Rockwell Manufacturing Company's ER industrial meter bulletin (OG-400) offers enough additional information on factors affecting meter selection to enable single-plant operators to be self-sufficient in ordering. The bulletin describes Rockwell's complete line of oscillating piston meters, recommended for a variety of chemicals and industrial liquids. Write Meter & Valve Division, Rockwell Mfg., 400 N. Lexington Ave., Pittsburgh 8, Pa.

A 16-page booklet, "*JAGUAR*, GUAR GUM by Stein Hall," describes the unusual combination of physical and chemical properties possessed by this new, versatile, pure vegetable colloid. Included is information on FDA standards concerning guar gum, methods of handling, types available, applications, etc. Write *JAGUAR* Dept., Stein Hall & Co., Inc., 285 Madison Ave., New York 17, N. Y.

"How to Solve Your Bread Wrapping Problem," new 20-page technical manual for bakers and wrapping machine operators, deals with proper handling and storage of waxed paper, installation and maintenance of wrapping machine, and correct machine operation. For copy, write Laurence T. Herman, executive director, Waxed Paper Merchandising Council, 38 S. Dearborn St., Chicago 3, Ill.

High power ultrasonic generators and transducers adaptable to practically any type of tank configuration and dimension are described in free Bulletin DR-2000, published by Acoustica Associates, Inc., Glenwood Landing, L. I., N. Y. The bulletin provides details of how 2 Acoustica generators, Model DR-2000AL (low frequency) and Model DR-2000AH (high frequency) can be applied for powering large scale, high volume batch or automated ultrasonic cleaning, machining, and liquid processing systems.

Crescent Metal Products, Inc., Cleveland, Ohio, announce a new 4-page Composite Catalog No. 203A showing typical units of the complete Cres-Cor line of aluminum food service equipment. The Catalog lists all the standard pan and tray sizes along with their actual dimensions.

Dow Corning Silicone Defoamers are described in a new bulletin available from Dow Corning Corp., Midland, Mich.

"Industrial Research Newsletter," November 1956, published by Armour Research Foundation of Illinois Institute of Technology, Technology Center, Chicago

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Standard of Quality for 30 years

BETTER JAMS
Jellies and Confections Because...

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• NUTRL-JEL Regular and Slow Set. } For Preserves, Jams, Jellies and Marmalades
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For full information, technical advice, and formulas write Dept. FT-2

Plants in Apple Regions From Coast to Coast Assure Dependable Supply
SPEAS COMPANY, GENERAL OFFICES, KANSAS CITY 20, MISSOURI

16, Ill., reporting on the recent Atomic Industrial Forum held in Chicago, points out that between 1955 and 1958 one-fourth of nuclear research and development outside the weapons area will be conducted by private industry. By 1964, cumulative spending by industry and government for nuclear research may hit $7.5 billion.

. . . *meeting hall and conference room*

A 3-day educational forum on instrumentation in the food industries, the third annual program of its kind, is scheduled for March 18-20 at The Foxboro Company, Foxboro, Mass. Forum sessions will cover electronic, mechanical, and pneumatic measurement and control, with special attention given to quality control and analytical measurements. Discussion of specific applications will occupy an entire session, and periods have been set aside for design considerations on centralized control panels and data logging. The group will have opportunity to preview new instrument designs and developments. The program will be of particular interest to processors of beet and cane sugar, grain products and corn products, as well as brewers, confectioners, canners and packers, and food equipment manufacturers. Since forum openings are limited, attendance will be confined to food men primarily concerned with profitable use of instruments. No charge is made for the 3-day sessions. Applications should be made to W. S. Young, manager, Food Industries Division, The Foxboro Company, Foxboro, Mass.

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IN-PLANT CHLORINATION

From start to finish of your processing operation, *Wallace & Tiernan In-Plant Chlorination* will keep your sanitation problems to a minimum by:

Eliminating slime formation on equipment and in work areas

Retarding bacteria growth

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The Sixth Annual Cottonseed Processing Clinic will be held February 4-5, 1957, at the Southern Regional Research Laboratory, 1100 Robert E. Lee Blvd., New Orleans, La., and is open to all persons interested in cottonseed processing. There will be sessions on "Industry Trends and Research Needs," and "Processing and Product Quality."

Early plans for the 48th annual meeting of the American Oil Chemists' Society, to be held at the Roosevelt Hotel, New Orleans, La., April 29-May 1, 1957, tentatively call for two symposia of invited papers and six sessions devoted to general papers. Correspondence may be entered into with Dr. O'Connor at SRRL. Abstracts of 100-300 words must be submitted to him by February 15.

The new Food Establishment Sanitation Advisory Committee of the Public Health Service held its first meeting November 27-29 in the Department of Health, Education, and Welfare Building in Washington, D. C. Of the 16 members who constitute the committee appointed by Surgeon General Leroy E. Burney, 5 represent national public-health associations; 4, State and local health organizations; 2, educational institutions; and 4 represent the National Restaurant Assn., National Licensed Beverage Assn., and the American Hotel Assn. During the November meeting, the Committee reviewed and evaluated the applicability of food and beverage sanitation procedures in the light of modern industry practices and a rapidly changing technology. Future work will include technical consultation and guidance to the Service in development of modern sanitation standards for restaurants and other food and beverage service establishments, for use by interested States and municipalities. This will serve as a means of providing a uniform approach to the service of safe foods and beverages to the public. John D. Faulkner, chief of the Milk and Food Program in the Public Health Service Division of Sanitary Engineering Services is Committee chairman.

. . . *building up and branching out*

Approval of the purchase of the assets of The Visking Corporation by Union Carbide and Carbon Corp., 30 E. 42nd St., New York 17, N. Y., was voted December 19 by Visking stockholders and became effective December 31, 1956. Terms of the agreement, previously approved by the directors of Union Carbide, call for the exchange of one share of Union Carbide stock for each 2½ shares of Visking stock currently held. A new Division of Union Carbide will be set up, to be known as Visking Company. Visking, founded in 1925, makes food casings used by packers for processed meat and sausage products, as well as polyethylene film and tubing important in the packaging industry. The 3000-employee company pioneered the use of cellulose meat casings. Union Carbide's major fields of interest are alloys and metals, carbon

products, industrial gases, chemicals, plastics, and nuclear energy.

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G. C. Pound, president of Kraft Foods Co., Chicago, has issued a denial of published reports that Kraft plans a new $5 million citrus processing plant at Lakeland, Fla. Mr. Pound said that Kraft has recently acquired a parcel of land near Lakeland which will be the site of a truck terminal and feed mill. The company now operates a citrus processing and packing plant at another site in Lakeland.

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Announcement has been made of the opening of a new extract and essential oil firm, Quintessence Laboratories, located at 6 Varick St., New York 13, N. Y. The new flavor firm is fully equipped to offer a complete line of flavoring extracts, essential oils, true fruit and artificial flavors, aromatic chemicals, and certified colors to the confectionery and bottling trade, as well as to the food and beverage industry in general. The organization is headed by Dr. Norman C. Larsen, previously associated in various responsible research and technical administrative capacities with Norda Essential Oil and Chemical Co., Polak & Schwarz, Inc., and Stuart Brothers, Ltd. He is a senior member of ACS, a professional member of IFT, and holds membership in the New York Association of Food Technologists, and AAAS. The new firm presently is setting up plans for the expansion of sales territories, which now are organized to cover only the area east of the Mississippi.

. . . Quintessence's Larsen

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Effective January 1, Hagan Corp., 323 Fourth Ave., Pittsburgh 22, Pa., changed its name to Hagan Chemicals & Controls, Inc., and merged its subsidiaries, Calgon, Inc., Hall Laboratories, Inc., and The Buromin Company, into the parent firm. Calgon and Hall will continue as divisions. The new name is intended to better describe the company's activities in the fields of chemical engineering and automatic control equipment, while the merger of the subsidiaries is expected to give the company some important tax advantages and operating economies. Best known Hagan chemical product is Calgon, a household water conditioner which has important industrial applications. Hagan controls and instrumentation systems are used in industrial operations depending on combustion, in aeronautical and automotive testing facilities, and in basic process control applications. The company recently expanded its line of industrial products to include a mechanical-type dust collector.

◆ ◆ ◆

Minneapolis-Honeywell Regulator Company has purchased Davies Laboratories, Inc., Beltsville, Md., manufacturer of specialized high-speed data recording systems. Activities of the Davies Laboratories will be integrated with those of Honeywell's Industrial Division, Wayne & Windrim Aves., Philadelphia. The

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PROFIT!
Long hours in the laboratory create short cuts to profit
The painstaking research and concentrated effort of Polak & Schwarz flavor specialists pay off at the point of sale, when the customer comes back for seconds and thirds.
Flavor superiority is well rewarded in dollars and cents.
Take the first step toward a superior product and your profit building program by requesting free samples.
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667 WASHINGTON ST., NEW YORK 14, N.Y.
Offices: Chicago, Ill., Los Angeles, Calif.; Toronto, Can.; Plants: Tetersboro & Guttenberg, N.J.; Culver City, Calif.; New York, N.Y.

Davies firm employs approximately 125 people, about one-fourth of whom are engineers and scientists.

Monsanto Chemical Company's Plastics Division has joined forces through a distributorship agreement with Plax Corp., Hartford, Conn., to develop and expand U. S. Markets for Plax's Polyflex 100 film and sheeting, used extensively in food packaging. The marketing agreement, announced jointly by R. F. Elder, president of Plax, and R. K. Mueller, vice president of Monsanto and general manager of its Plastics Division, is described as an unusual marketing plastic film and sheeting; and Monsanto is its own sales and development activities with the resources of Monsanto's large sales and development organization and over 60 years of experience in marketing concept whereby Plax is able to supplement able to expand the range of products it offers to its many customers. The end result is expected to be a substantial broadening of the market for plastic materials.

Beckman Instruments, Inc., Fullerton, Calif., has announced acquisition of Watts Mfg. Co., Inc., Ronceverte, W. Va., manufacturer of a new, continuous-action gas chromatograph. Watts also manufactures temperature test stands for jet engine development and automatic flaw detectors for the textile industry. The new gas chromatograph was developed by the Special Instrumentation Department of Carbide and Carbon Chemicals Co., a division of Union Carbide and Carbon Corp., at South Charleston, W. Va., in cooperation with Watts Mfg. Co. It is being manufactured under license from Union Carbide and Carbon Corp. J. Stuart Watts, founder-president of Watts Mfg. Co., Inc., will remain on the staff as resident manager of the Ronceverte plant.

The new Johns-Manville synthetic silicates plant at Lompoc, Calif. is now in full production making an initial line of 4 synthetic calcium silicates marketed under the name, Micro Cel. These serve as absorbents and anti-caking agents to improve the free-flowing characteristics of insecticides, cleansers and detergents, fertilizers, and certain food and feed products. Additional calcium silicates are under development, and Johns-Manville also is working on magnesium and other metallic silicates. Heart of the new plant is the group of 4 reactors within a maze of pipes (middle foreground) where slurries of lime and diatomaceous silica are combined chemically to make a calcium silicate. The plant is largely automatic in operation, and present capacity is upwards of 1,000 carloads a year.

Highlight of 1956 for Celanese Corporation of America's Plastics Division was the start of commercial production of Fortiflex, a polyolefin with a combination of rigidity, toughness, heat resistance, and chemical inertness that is expected to have widespread applications in housewares, containers, wire and cable covering, closures, and other fields. Full-scale production at an annual rate of 40 million pounds is expected by the first part of 1957 at the company's new Houston plant.

. . . *the human element*

Appointment of IFT member **Harold L. Janovsky** as chief flavor chemist, replacing Dr. Edmund H. Hamann whose retirement was effective January 1, 1957, was announced by Fritzsche Brothers, Inc., New York City. Mr. Janovsky, who comes to the Fritzsche organization after almost a year's service as director of the Flavor Division of Dodge & Olcott, Inc., has an industry background of 22 years in the chemistry of beverage and food flavorings and essential oils. He is a member of ACS, Flavoring Extract Manufacturers of the U. S., and the Society of Flavor Chemists. Prior to his D&O affiliation, he held technical and executive positions with Virginia Dare Extract Co., Seeley & Co., Inc., and Aroscent, Inc.

The 1956 Southern Chemist Award given annually in recognition and honor of distinguished service to the profession of chemistry in the South, was presented on December 7 to **Dr. C. Harold Fisher**, chief, Southern Utilization Research Branch, USDA, New

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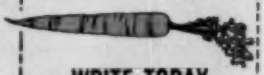

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Orleans, La. Occasion was the banquet of the Southeastern Regional meeting of ACS at the Peabody Hotel in Memphis, Tenn. Dr. Fisher was cited as "a brilliant research scientist and inspiring and untiring leader of research . . . (who) has brought an informed chemical approach to the solution of Southern agricultural problems which has opened up new horizons for research, and developed new concepts for furthering agriculture in this research."

◆ ◆ ◆

Edwin Stein, chairman of the board of Stein, Hall & Co., Inc., 285 Madison Ave., New York 17, N. Y., was honored on December 12 at a testimonial dinner in celebration of his 40th anniversary of service with the company. The firm recently announced appointment of **Emil Alter** as head of its Natural Gums Department at Long Island City, N. Y., replacing **Arthur Goldstein,** recently named chief chemist of the company's Paper Laboratory, and appointment of **Richard J. McKiernan,** most recently chief chemist in the Industrial Food Laboratory, as chief chemist of that Laboratory.

. . . JAGUAR'S Alter

. . . Friends, Romans . . . (Bob) Countryman!

◆ ◆ ◆

Appointment of **R. C. (Bob) Countryman** to the sales staff of Modern Packages, Inc., Division of Standard Packaging Corp., 551 Fifth Ave., New York 17, N. Y., was announced recently. Since 1949 he had been with Shellmar Products Corp., now the Shellmar-Betner Division of Continental Can Co.

◆ ◆ ◆

John F. Ovsik has been promoted to director of purchases for Vulcan Containers Inc., Bellwood, Ill., and as such will be responsible for buying steel, paint, and other materials used by the firm in manufacturing the widest variety of pails in the steel shipping container industry.

◆ ◆ ◆

. . . Mr. Smith goes to Hoopeston!

Food Machinery and Chemical Corporation has announced promotion of **Marion O. Smith,** a 20-year FMC veteran, to plant superintendent of the Canning Machinery Division's Eastern Operation at Hoopeston, Ill., succeeding Robert Jabbusch who has resigned to accept a position in Milwaukee, Wis. In his new capacity, Mr. Smith will have direct supervision of all manufacturing operations in the Hoopeston plant.

Dr. James G. Miller has joined the staff of Arthur D. Little, Inc., Cambridge, Mass., following a 3-year post-doctorate investigation in pharmacology at the University of Michigan. His major interests will be in expanding ADL activities in the areas of pharmacology and toxicology. He also will be active in the present ADL programs in biochemistry, chemical carcinogenesis, clinical evaluation, and chemotherapy.

◆ ◆ ◆

Peter J. Metz has been promoted to superintendent of the Los Angeles plant of Western-Waxide Specialty Division, Crown Zellerbach Corp. Mr. Metz's most recent position was divisional technical superintendent of the Production Research and Development Laboratory at headquarters in San Leandro, Calif. He was responsible for the over-all quality control of Western-Waxide products.

. . . Western-Waxide's Metz

◆ ◆ ◆

Walter H. Goehring, newly appointed division manager of manufacturing, Can Division, Crown Cork & Seal Co., Inc., was one of the 50 recipients of the alumni honors award bestowed December 2 at Drexel Institute, Philadelphia. Mr. Goehring joined Crown in June as special assistant to the executive vice president of the company, after resigning as general factory manager of SKF Industries, Inc.

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The method of determining mixing properties of hard red spring wheat flours interests three visitors to the Department of Cereal Technology, North Dakota Agricultural College. From left are Castilla Chacon, cereal chemist from Mexico; Dr. Rae H. Harris, chairman of Cereal Technology, North Dakota Agricultural College; Gregario Vazquez, graduate student in plant breeding, also from Mexico, and Dr. Mustafa Ulnus, professor in the College of Agriculture at Ankara University, Turkey.

George B. Estes has been appointed manager of the Industrial Engineering Services Division of General Foods Corp., White Plains, N. Y.

During an intensive 2-day session of the Board of Directors of the National Confectioners' Assn., held at the Drake Hotel, Chicago, November 30 - December 1, **Robert B. Schnering** presented his resignation as chairman of the Board and chairman of the NCA Executive Committee, since he is not at present actively connected with the candy industry. **Victor H. Gies,** Mars, Inc., was elected to complete Mr. Schnering's unfinished term in the two posts.

Dr. D. M. Doty, IFT member, and assistant director of research and education of the American Meat Institute Foundation, Chicago, has been elected chairman of the ACS Division of Agricultural and Food Chemistry for 1957. He succeeds **Dr. Albert L. Elder,** also a member of IFT and director of research at Corn Products Refining Co., Argo, Ill. Dr. Doty and Elder will represent the Division on the national Society's Council.

Appointments of **W. D. McDonald** as vice president in charge of sales and **A. E. Watts** as vice president in charge of plant operations were announced by Sun Tube Corporation of Canada, Ltd., a subsidiary of Sun Tube Corporation of Hillside and Washington, N. J. Sun Tube is a leading manufacturer of collapsible metal tubes, impact extrusions, and aerosol valves.

Standard Brands, Inc., New York City, has announced that **O. Lester Applegate,** chief engineer of the company, was elected vice president in charge of engineering at the meeting of the board of directors on November 29. Mr. Applegate, most recently plant manager of the firm's Peekskill, N. Y., plant, has been with Standard Brands since 1942.

. . . Applegate of Standard Brands

Dr. Ralph M. Ennis has been appointed senior psychologist in the Psychometrics Department of the U. S. Testing Company's laboratories in Hoboken, N. J. He joins a team of psychologists and home economists who assist clients in food, clothing, appliance, and cosmetic industries in obtaining valid measurements of consumer preference by application of the methods of experimental science. Dr. Ennis has been a teacher of psychology and education at Hunter College, New York City, and is continuing lecture activities at Hunter College School of General Studies.

. . . psychologist Ennis of U. S. Testing

Polak's Frutal Works, Inc., manufacturers of fine Perfume and Flavoring raw materials, held their annual sales meeting on December 10th through December 12th at their plant in Middletown, N. Y. All U. S. A. and Canadian representatives attended this meeting. On December 11th the company gave a dinner party at the Orange County Golf Club for the

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executive, sales, and technical staff and their ladies. P. F. W.'s annual Christmas party for its personnel was also held at the Orange County Golf Club and a most pleasant evening was enjoyed by everyone.

◆ ◆ ◆

W. A. (Bill) Hoover, for the past several years assistant to the director of The Refrigeration Research Foundation, resigned in October to join the Corn Industries Research Foundation, Washington, D. C., as manager of the Technical Service Division.

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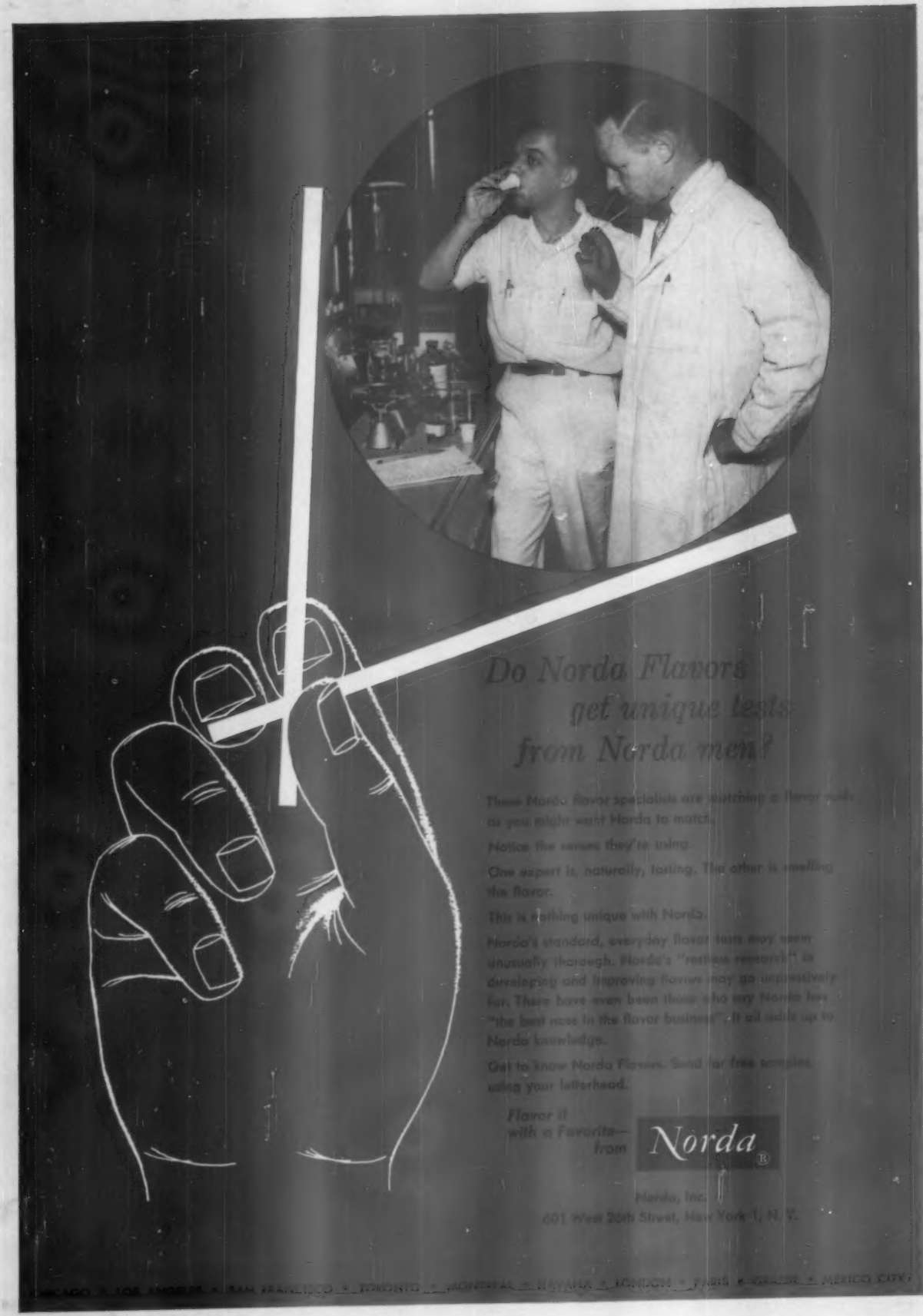

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